07:25:37 OCA PAD INITIATION - PROJECT HEADER INFORMATION 08/07/95

Active

Project #: D-48-X17 Cost share #: Rev #: 0
Center # : 10/24-6-R8628-0A0 Center shr #: OCA file #:
Work type : RES
Contract#: SUBCONTRACT SIGNED 7/24/95 Mod #: Document : SUBCONT
Prime #: DE-AC05-840R21440 Contract entity: GTRC

Subprojects ? : Y CFDA: NA
Main project #: PE #: NA

Project unit: DEAN ARCH Unit code: 02.010.170
Project director(s):
CIRCEO L JR DEAN ARCH (404)894-3390

Sponsor/division names: PLASMA ENERGY CORPORATION / RALEIGH, NC
Sponsor/division codes: 215 / 051

Award period: 950724 to 960723 (performance) 960723 (reports)

Sponsor amount	New this change	Total to date
Contract value	99,840.00	99,840.00
Funded	99,840.00	99,840.00
Cost sharing amount		0.00

Does subcontracting plan apply ?: N

Title: PLASMA PROCESSING OF SURROGATE THORIUM NITRATE CRYSTALS

PROJECT ADMINISTRATION DATA

OCA contact: Ina R. Lashley 894-4820

Sponsor technical contact

DR. BUD CAMACHO, PRESIDENT & CEO
(919)676-5304

PLASMA TECHNOLOGY CORPORATION
8601 SIX FORKS ROAD, SUITE 400
RALEIGH, NC 27615

Sponsor issuing office

DR BUD CAMACHO, PRESIDENT & CEO
(919)676-5304

PLASMA TECHNOLOGY CORPORATION
8601 SIX FORKS ROAD, SUITE 400
RALEIGH, NC 27615

Security class (U,C,S,TS) : U ONR resident rep. is ACO (Y/N): N
Defense priority rating : NA NA supplemental sheet
Equipment title vests with: Sponsor X GIT
PER STATEMENT OF WORK, P. 2, TASK 2
Administrative comments -
INITIATION OF ONE-YEAR SUBCONTRACT UNDER DOE PRIME CONTRACT. CAPPED OVERHEAD.
*NOTE: PROJECT DIRECTOR TO NOTIFY OCA/PAD 60 DAYS PRIOR TO 75% EXPENDITURE.**

U

2-N-SR313

GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

NOTICE OF PROJECT CLOSEOUT

Closeout Notice Date 11/11/96

Project No. D-48-X17_____ Center No. 10/24-6-R8628-0A0_

Project Director CIRCEO L JR_____ School/Lab DEAN ARCH_____

Sponsor PLASMA ENERGY CORPORATION/RALEIGH, NC_____

Contract/Grant No. SUBCONTRACT SIGNED 7/24/95____ Contract Entity GTRC

Prime Contract No. DE-AC05-840R21440_____

Title PLASMA PROCESSING OF SURROGATE THORIUM NITRATE CRYSTALS_____

Effective Completion Date 960723 (Performance) 960723 (Reports)

Closeout Actions Required:	Y/N	Date Submitted
Final Invoice or Copy of Final Invoice	Y	_____
Final Report of Inventions and/or Subcontracts	Y	_____
Government Property Inventory & Related Certificate	Y	_____
Classified Material Certificate	N	_____
Release and Assignment	Y	_____
Other _____	N	_____

Comments_____

_____

Subproject Under Main Project No. _____

Continues Project No. _____

Distribution Required:

Project Director	Y
Administrative Network Representative	Y
GTRI Accounting/Grants and Contracts	Y
Procurement/Supply Services	Y
Research Property Managment	Y
Research Security Services	N
Reports Coordinator (OCA)	Y
GTRC	Y
Project File	Y
Other _____	N
_____	N

NOTE: Final Patent Questionnaire sent to PDPI.

D-48-X17
#1
(new)

Georgia Tech
RESEARCH INSTITUTE

Georgia Tech Research Institute
Georgia Institute of Technology
Atlanta, Georgia 30332-0800
USA

November 15, 1995

Dr. Bud Camacho
President & CEO
Plasma Technology Corporation
6601 Six Forks Road Suite 400
Raleigh, NC 27615

Dear Bud:

The quarterly process report (QPR) for July 24, 1995 to October 31, 1995 for the Plasma Processing of Surrogate Thorium Nitrate Crystals is attached. This QPR is delivery item #1. Please contact me for additional information as required.

Sincerely,

Robert (Bob) A. Newsom, Research Technologist II
Manager, Plasma Application Research Facility
Georgia Institute of Technology, Georgia Tech Research Institute
Electro-Optics, Environment, and Materials Laboratory
Environmental Engineering Branch
151 Sixth St. O'Keefe Bldg., Atlanta, GA. 30332-0837
404-894-8047, Fax 404-894-2184

QUARTERLY PROGRESS REPORT

Deliverable:	**#1**
Title:	**Plasma Processing of Surrogate Thorium Nitrate Crystals PTC Contract for Prime :#DE-AC05-840R21440**
Department(s):	Georgia Institute of Technology
Main #:	D-48-X17: Architecture Construction Research Center
Sub(s) #:	A-5017-000 Georgia Tech Research Institute
Contract Duration:	12 months: July 24, 1995 to July 23, 1996
Reporting Period:	**July 24, 1995 to October 31, 1995**
Date Due:	November 15, 1995
Prepared by:	Robert A. Newsom

Introduction

Plasma Technology is being developed at the Georgia Tech Plasma Application Research Facility (PARF). Various programs are currently being investigated for the improved operation and processing of plasma applications. The programs consist of using the plasma torch for research on asbestos destruction, municipal waste processing, incinerator ash processing, soil remediation, soil stabilization, and surrogate testing of both hazardous and low-level radioactive wastes. Plasma Technology Corporation (PTC) is developing system designs and implementations for processing materials for various organizations including the Defense Logistics Agency (DLA). The DLA materials requiring treatment includes thorium nitrate crystals.

The Plasma Application Research Facility at Georgia Institute of Technology will provide testing and engineering support for the development activities by Plasma Technology Corporation for these efforts. The program participants at Georgia Tech will consist of the Construction Research Center (GT/CRC) and the Georgia Tech Research Institute (GTRI).

Project Summary & Goals

The initial tasks for this program will consist of the treatment of thorium nitrate crystals. A test plan is to be generated, procurement of materials will be conducted, a system design criteria will be established, and the conduction of tests will be performed. The conduction of tests includes data collection, sample collection, data analysis, and reports. The program will use a team approach with collaboration between the government, industry, PTC and Georgia Tech staff members. The program will advance the processing and development of plasma technology for degassing and vitrification of materials specifically thorium nitrate crystals A surrogate of thorium nitrate will be used at the Georgia Institute of Technology Plasma Application Research Facility.

Report Period Accomplishments:

1. This program was initiated in March 1995 as a feasibility for plasma processing of the DLA thorium nitrate. A draft for a surrogate test program was initiated and a meeting was held in Knoxville, TN on May 18, 1995 to discuss the concept. The following is trip report for the meeting:

   Trip Report for Meeting in Knoxville on May 18, 1995 for discussions concerning the Surrogate Test Program being proposed for the disposal of Thorium Nitrate by Plasma Processing. This test program is being supported by:

   Mr. F. Kevin Reilly
   Environmental Protection Specialist
   Defense Logistics Agency
   1745 Jefferson Davis Highway
   Crystal Square #4 Suite 100
   Arlington, Virginia 22202

   Meeting Attendees: 8 AM to 3 PM

Name	Organization	Phone
Frank Van Ryn	LMES	615-574-1907
Doug Hoffman	LMES	615-574-3896
L. A. Harris	LMES	615-574-5127
Doug Laird	ScienceVentures	619-242-7354
Bob Newsom	Georgia Tech	404-894-8047
Bill Hermes	LMES	651-574-9568
S. L. (Bud) Camacho	PTC	919-676-5304
Terry L. Sams	LM	615-241-2409
Alexander L Suris	Moscow State Academy of Chemical Engrg	7 095-267-07-92

   The meeting was started with introductions and organizational functions from each attendee.

   The purpose of this meeting was to discuss the surrogate test program at Ga Tech and the overall program plan for processing the Thorium Nitrate materials for disposal by DLA.

   Bud Camacho gave a update on the system being installed and operated for DLA to process asbestos materials. (Note: Bud requested that Ga Tech/GTRI quote a video view port monitor system to be fabricated and installed for process video recording).

   Bill Hermes gave an overview of the surrogate test plan

- Materials selection

- GT PARF system configuration

- Design Considerations Original concept for submerged torch processing vs semi-batch drum feeding, tapping design.

A general open meeting discussion on these and additional items was identified to be discussed at this meeting.

Additional items:
- sample configuration and feeding
- safety and health issues
- Dr. Suris inputs
- product testing and criteria
- test system mods (fan, leakage, etc)

Discussions:

The surrogate test plan
- close compatibility to thorium nitrate (phase relationships were discussed. Dr. Suris gave results of his data.
- question on using cerium in the surrogate
- question of using two different sample makeup's

Bill Hermes will have responsibility for final mixture
- Offgas as primary surrogate testing purpose Vs product analysis
  *Decided gas measurement was a primary purpose of the tests
- torch movement for degassing during testing
- melting torch position
- use of argon as gas medium
- *Decided that torch movement will be applied during the tests, argon will not be used to make testing procedures simpler at this time.

Terry Sams joined meeting to discuss the process for approval of potential disposal sites for the environmental restoration waste streams- possible use of disposal at Nevada Test Site of processed material. Question on the original use of thorium nitrate as a defense item? Discussion on the process and certification of the material and the length of time 1-year to 3 years for this process. Cost for disposal sites and transportation will be looked into for total program evaluation Vs alternative disposal.

Pilot Scale Design Discussions:
- non-transferred Vs transferred
- solid form oxidation
- weight reduction
- volume reduction
- rate of injection feeding/tapping
- thermal measurements

Surrogate test schedule

Depends on having a contract iin plane before starting work. This contract would be from DLA to Lockheed Martin to Plasma Technology Corporation to Ga Tech. It would cover the thorium nitrate program and possible assistance on the asbestos system.

Bob Newsom prepared for Bud Camacho a format for putting together a SOW to GT. This program needs to be TASK oriented. The current tasks identified include:

Task 1. Surrogate Test Plan and Preparation for TEST (4-tests)

Task 2. Additional Test (if needed)

Task 3. Design Engineering Support

Task 4. Site Engineering Support

Task 5. Final Program Report

Task 6. NLA System View Port Design (both systems)

The schedule agreed upon at the meeting was for every effort to have a contract in place by the first of June. In June the start of materials be procurement, modification and preparation of the test system will begin, and sample prep started. The first test run start would be the middle of July. The goals is to have the four tests completed by the end of August. This schedule is dependent on the start of the contract and availability of materials and staff.

Contract issues to be finalized include:
- Type of contract Fixed Vs Cost Reimbursement
- Duration (recommend 2 year task oriented)
- Deliverable (qtrly & one final report)
- Patent rights
- Budget for each task

Action Items:

1. Bud Camacho needs to get a SOW to GT for contract initiation.

2. Bill Hermes will rework and update test plan for final review.

3. Bob Newsom will get cost and schedule for GT Tasks.

4. Terry Sams will investigate disposal criteria

5. Frank Van Ryn will inform Kevin Reilly on schedule and funding.

6. Lockheed Martin program staff will visit asbestos processing system 5/24

2. A second meeting to finalize the test plan was held in Raleigh, NC at PTC on June 20, 1995. The meeting attendees were:

Name	Organization	Phone
Frank Van Ryn	LMES	615-574-1907
Doug Hoffman	LMES	615-574-3896
Hom-Ti Lee	K-25	615-574-1603
Doug Laird	ScienceVentures	619-242-7354
Bob Newsom	Georgia Tech	404-894-8047
Bill Hermes	LMES	651-574-9568
S. L. (Bud) Camacho	PTC	919-676-5304
Steve Cockrell	PTC	919-676-5354

The test plan was discussed with inputs from all parties. The document is #ES/CET-95-1 dated June, 1995.

3. The contract with PTC was finalized on July 24, 1995 with Georgia Tech to provide support for this test program.

4. The schedule for the surrogate test plan was arranged by Bob Newsom and Bill Hermes for:

- August 27, 1995 to September 5, 1995: Test set up (gas collection)
- September 5, 1995- to September 10, 1995 Hot Testing no sample
- September 10, 1995 to September 21, 1995 Test Runs
- September 21, 1995- September 29, 1995, Test Clean up and Data Analysis
- Month of October, 1995 Data Analysis and Design Criteria.

5. A paper on this test program was presented as a poster session and published in the proceedings for the 2nd International Symposium on Environmental Technologies-Plasma Systems and Application held October 8-12,1995 in Atlanta, Georgia. Members of the test team attended and participated in the symposium. The paper is a result of the test program and is attached.

Next Period Plans: November 1, 1995 to January 31, 1995

1. The final clean up and data analysis for the surrogate testing will be performed. The crucible and gas sample tube will be returned to Georgia Tech by Lockheed-Martin (K-25). The team approach for the test report will be compiled.

2. A meeting is scheduled for November to report the results and to discuss the next t phase for system development. This meeting is currently planned to be located

PLASMA PYROLYSIS VITRIFICATION (PPV) TECHNOLOGY DEMONSTRATION PROGRAM AS APPLIED TO THE DEFENSE NATIONAL STOCKPILE OF THORIUM NITRATE

W.H. Hermes, D.P. Hoffmann,
L.V. Gibson, F. Van Ryn
Oak Ridge K-25 Site‡
Oak Ridge, TN 37831-7272

L.A. Harris
Midwest Technical, Inc[1]
Oak Ridge, TN 37831

H.T. Lee
Oak Ridge Associated Universities[2]
Oak Ridge, TN 37831

S.L. Camacho
Plasma Technology Corporation[3]
Raleigh, NC 27624-8356

L.J. Circeo
R.A. Newsom
Georgia Institute of Technology[4]
Atlanta, GA 30332

D. Laird
Science Ventures Inc.[4]
San Diego, CA 92123

A.L. Suris
Moscow Academy of Chemical Engineering[4]
Moscow, Russia

‡Managed by Lockheed Martin Energy Systems, Inc., for the U.S. Department of Energy.
[1]Under subcontract 1BK-02268C with Lockheed Martin Energy Systems, Inc.
[2]Under subcontract 19KAGB85V with Lockheed Martin Energy Systems, Inc.
[3]Under subcontract 91K-HDT01V with Lockheed Martin Energy Systems, Inc.
[4]Under subcontract with Plasma Technology Corporation

OVERVIEW

Thorium (^{232}Th) can be utilized as fuel in breeder style nuclear reactors. ^{232}Th is considered a fertile nuclide since it can be fissioned by high energy (> 1 MeV) neutrons to produce fissile uranium (^{233}U). The Defense National Stockpile Center (DNSC) currently has ^{232}Th in storage in the chemical form of thorium nitrate, $Th(NO_3)_4 \cdot 4H_2O$. The potential use of ^{232}Th is being phased out and hence there is a need to mitigate any potential future environmental or exposure hazards associated with this material. Since ^{232}Th has a long half-life ($1.41x10^{10}$ yrs.), the current program is evaluating treatment methodologies to safely produce the less leachable oxide. The more chemically stable oxide will be lower in mass and volume and exhibit low leachability characteristics making it optimized for licensed long-term storage. This paper will discuss some work completed and the experiments which are planned for completion at the Ga Tech Plasma Application Research Facility (PARF). This paper will also present the types of experiments being considered using actual thorium nitrate and how they will be used to support the evaluation of plasma processing application to the thorium nitrate inventory. The experiments at Ga Tech are planned for September 1995 and will utilize non-radiological surrogates for the thorium oxide. Although the early publishing date of this paper preclude the incorporation of any actual results, the planned poster session will include all verified data collected to date.

THORIUM NITRATE INVENTORY/DEMONSTRATION

The DNSC currently stores 2,596 short tons (1,814 metric tons) of thorium nitrate in the Curtis Bay, MD facility and 952 short tons/864 metric tons in the Hammond, IN facility. The thorium nitrate is stored in various size drums with various internal configurations. The specific sequence of constituents within each drummed configuration is represented in Table I. The average drummed constituents and number of drums for each configuration is represented in Table II. The drum ID designates their storage location (MD = Maryland, IN = Indiana). The program is structured to evaluate the safety, hazards, and process design criteria in transforming the thorium nitrate to a low volume, stable final form. Due to the considerations of thoria dust formation and potential for violent exothermic reactions, the process design basis is comprised of a two stage process: low-temperature (600°C/873°K) denitration/dehydration followed by high temperature plasma vitrification.

A drum scale demonstration is planned for completion at the Oak Ridge, Tennessee K-25 Site in 1996. A building at the K-25 site has been prepared to house this demonstration. The expected criteria for evaluating the acceptability of this plasma vitrification process are:

- Significant volume reduction and weight reduction
- Radon and radioactive constituents are controllable and accountable
- Whole drums can be processed to accomplish the sequence of dehydration, denitration, and vitrification.

TABLE I. DNSC THORIUM NITRATE PACKAGING CONFIGURATIONS

Curtis Bay	Package 1 - Thorium nitrate inside a polyethylene bag, inside a 20 gallon fiber drum, inside a 10 mil polyethylene bag, slaked lime inside a 30 gallon cross linked polyethylene drum, inside a 35 gallon metal drum Thorium nitrate inventory - 15,701 drums - 1,570 short tons (1,424 metric tons) Package 2 - Thorium nitrate in polyethylene bag inside 55 gallon metal drum Thorium nitrate inventory - 2,597 drums - 910 short tons (825 metric tons) Package 3 - Thorium nitrate inside polyethylene bag, inside 30 gallon painted drum, inside a 10 mil polyethylene bag, slaked lime inside a cross linked polyethylene drum liner, inside a 55 gallon metal drum painted with a epoxy/phenolic resin (.6-.7 mil thickness) Thorium nitrate inventory - 184 drums - 18 short tons (16 metric tons) Package 4 - Thorium nitrate inside fiber board drum, inside 10 mil polyethylene bag, slaked lime inside 40 gallon crossed linked polyethylene drum. Drum lid is bolted sand sealed to prevent moisture using a polybutyllen butyl glazing tape Thorium nitrate inventory - 753 drums - 75 short tons (68 metric tons) Package 5 - Thorium nitrate polyethylene bag inside, inside a 55 gallon drum, slaked lime inside a 85 gallon painted metal drum Thorium nitrate inventory - 66 drums - 22 short tons (20 metric tons)
Hammond, IN	Package 1 - Thorium nitrate inside a polyethylene bag, inside a fiber drum, inside a 55 gallon drum, vermiculite inside a 70 gallon painted metal drum Thorium nitrate inventory - 2,308 drums - 952 short tons (864 metric tons) *NOTE: There are 4 bricks and about 3 inches asphalt in the bottom of the 70 gallon overpack to support the 55 gallon inserted drum. The void space between the drums is filled with vermiculite or similar material*

TABLE II. THORIUM NITRATE STORAGE DRUM CONFIGURATION

Item	MD-1	MD-2	MD-3	MD-4	MD-5	IN-1
	Amount of components in drums (lbs) (1 Kg = 2.2 lb)					
$Th(NO_3)_4*4H_2O$	200	726	200	200	663	825
$Ca(OH)_2$	44	0	44	105	269	0
Polyethylene	30	1	39	34	1	22
Fe	48	60	60	0	152	139
Al_2O_3	0	0	0	0	0	63
Asphalt	0	0	0	0	0	43
Drum Size (Liter)	132.5	208.2	208.2	151.4	321.7	265
Drum Size (gal)	35	55	55	40	85	70
# of Drums	15,701	2,597	184	753	66	2308

- Leachability of the vitrified oxide (final product) is suitable for safe, long-term storage
- The process meets environmental health and safety standards

The low temperature stage will remove the nitrate and water from the system and convert the thorium nitrate hydrates to thoria (ThO_2). The low temperature stage should also convert the calcium hydroxide to calcium oxide and the polyethylene (or asphalt) to carbon (char) and carbon oxides. The iron drum and the alumina are not expected to be greatly effected by the low temperature process. The results of the drum scale demonstration will be used to ascertain the need to degas the drum matrix in a vessel separate from the plasma pyrolysis vitrification (PPV) reactor.

The residuals in the drum from the low-temperature process (e.g., Fe, ThO_2, CaO, C, Al_2O_3) will then be processed by plasma pyrolysis vitrification (PPV). The objective of the high-temperature stage is to maximize the density of the final product and help to encapsulate and/or integrate the thoria into a glass or slag matrix.

The thorium in its present configuration will have significant radon (^{220}Rn) gas collected within the crystalline structure. The radon gas is from the ^{232}Th decay. The radon gas will be released as the drum is vented and the thorium is heated. ^{220}Rn will be collected in the air pollution control (APC) carbon adsorption unit, HEPA filters/aqueous scrubber stream. Thorium nitrate hydrates have a variable water content, cake very badly, and are difficult to meter or transfer as a solid. Transfers of whole, unopened drums were adopted for the baseline process design to minimize pretreatment/handling requirement and take full advantage of the high temperature plasma heating process.

THERMAL DATA ON DRUM CONSTITUENTS

THORIUM NITRATE (TN) HYDRATES. Data in the "Gmelin Handbook of Inorganic Chemistry" lists tetrahydrate, pentahydrate and hexahydrates of thorium nitrate as solid phases. One phase study of the $Th(NO_3)_4 \cdot XH_2O$ system revealed the melting temperatures of the tetrahydrate and hexahydrate at ~150°C (423°K) and ~110°C (383°K), respectively.

Thermogravemetric (TGA) data for $Th(NO_3)_4 \cdot 5\ H_2O$ heated in air at 6°C/min shows the final temperature of decomposition to be between 400-500°C (letter memo comments on the Decomposition Behavior of Thorium Nitrate Hydrates, P.A. Haas to W.H. Hermes, 11/11/94). The T.G.A. shows a change of slope occurring before ~200°C (473°K) with initial weight losses probably as water and possibly corresponding to the formation of such hydrates as $Th(NO_3)_4 \cdot 3\ H_2O$ in air which reportedly forms between 118-144°C on heating $Th(NO_3)_4 \cdot 4\ H_2O$ in air. Following the change of slope there appears to be a constant weight loss up to ~300°C (573°K) where the curve flattens which could correspond to the phase $ThO(NO_3)_2 \cdot 0.5\ H_2O$. It should be noted that it is well documented that increased heating rates shift reaction temperatures to lower values. Rapid heating of either surrogate or real drums will affect reaction temperatures and kinetics.

Based on a literature search completed, a series of steps that occur on heating hydrated thorium nitrate directly in a plasma torch, were postulated. The reaction steps are as follows:

1. The solid melts to yield a solution of medium viscosity.

2. The solution becomes more viscous with the loss of water but with minimal loss of nitrates.

3. The losses of nitrate and water converts the material to a very thick melt and then to a slag or glass.

4. Continued change sees the solid becoming a porous ThO_2 agglomerate with a relatively low bulk density, perhaps as low as only 20% of theoretical density.

5. The agglomerates have been shown to be in the range of 20-30 microns but may contain even smaller thoria crystallites. Attempts to form dense thoria by thermal denitration were aided by small additions of Al_2O_3 or CaO. This latter compound will be present in much larger amounts as a constituent in the drum.

Ca(OH)$_2$. $Ca(OH)_2$ supposedly interfaces with the thorium nitrate separated by a plastic surface in several of the drum configurations. Literature values for the thermal decomposition of $Ca(OH)_2$ are listed as ~600°C (873°K). However, possibilities exist for at least partial conversion of $Ca(OH)_2$ to probably $CaCO_3$ which has a substantially higher temperature of melting. In addition, it is known that ThO_2 + CaO can react to form $ThCaO_3$ which melts at 2300°C (2573°K). Some reaction of these phases at the interface might be expected.

Fe. Drums that hold the mixture are most probably a low carbon steel. Years of exposure to ambient conditions have affected the exterior surface. Oxidation of the surface is probably minimal because of exterior paint nevertheless we need to recognize that possibility. The presence of iron oxides would not change the melting temperatures in a significant way since metal and oxides form melts at $\leq$1550°C (1823°K). An important question is how iron will act in a molten bath with other metal oxides, e.g., CaO, ThO_2 and possible glass formers.

ADDITIVES. Since ThO_2 is the most refractory oxide, it is envisioned that additives may be required to lower the fusion temperature of the molten bath. The formulation of a glass forming mixture should take advantage of the existing elements present (Ca, Fe). The minimum design basis temperature at which the bath should be maintained is selected to be 1550°C based on iron oxides and iron melting at near 1500°C (1773°K). One phase diagram reference (Nuclear Technology, T.S. Snidhar, 1985) showed the liquidus temperature for B_2O_3 - ThO_2 can be as low as 1500°C with 30 to 90 mole% of ThO_2 present in the system. The use of additives will be investigated to minimize bath temperature and control viscosity.

An alternate use of additives may be to suppress entrainment of thoria dust by enhancing adhesion and agglomeration of micron-size ThO_2 crystals. Such an additive could be introduced

in minor proportion before low temperature thermal denitration. It could reduce filter burden in both low and high temperature process steps. For example, boron forms an oxide eutectic with thorium melting at only 350°C (623°K). The eutectic forms before decomposition of thorium nitrate is complete, so that thoria particles are never free of the binding influence of liquids.

RADIOLOGICAL CONSIDERATIONS

The storage, handling, processing and transport of thorium (and associated radioactive progeny) will require radiological exposure monitoring as well as administrative and engineering controls to maintain exposures below regulatory limits and to levels that are as low as reasonably achievable (ALARA). Properly packaged, thorium nitrate compounds present only an external radiation hazard. However, associated with vitrification, thorium and radioactive progeny exposures are possible with products in both contained and uncontained forms. Further, non-radioactive elements which serve to attenuate radiation in thorium nitrate may be liberated or added and radiation levels from comparable masses of pre- and post-vitrification thorium compounds may differ. The demonstration will provide information necessary to develop a vitrification specific radiological protection program and a basis for comparison with radiological protection programs of alternative thorium nitrate treatment processes.

NATURALLY OCCURRING RADIOACTIVE MATERIALS. There are three naturally occurring radioactive decay series which may be found in and extracted from ores. They are denominated as the uranium series, the thorium series, and the actinium series. The thorium series is headed by the very long lived thorium-232. The series begins with thorium-232 that decays to radionuclides of radium, actinium, thorium, radon (a mobile, inert gas), polonium, lead, bismuth, thallium and ends with transformation to stable (i.e., non-radioactive) lead. In the absence of physical separation processes, an equilibrium is reached in which the number of atoms of each nuclide of the radioactive series that decays during a specific time interval nearly equals the number of decays of the parent nuclide in the series. The demonstration will provide information regarding the state of equilibrium in the series and the affects on that equilibrium by the vitrification process.

CHARACTERISTICS RELATED TO RADIATION DOSE. The potential radiation dose that workers or the public may receive from exposure to radioactive material (i.e., the thorium decay series) is determined by a number of factors. These include the amount of material involved, the types of radiation emitted by the material involved, the chemical and physical form of the material, the solubility of the material, the particle size distribution of the material, the duration of the exposure, the amount of material that may be resuspended from past releases (and ongoing operations), dispersion and dilution conditions at the time of exposure, the ingestion pathways involving contaminated water, food stuffs and animal feeds, and the demographic and physiological characteristics of the population exposed. The demonstration will provide information regarding these factors relative to the thorium nitrate vitrification process.

EXTERNAL RADIATION. External sources of radiation exposure to workers are caused by the concentrations and quantities of naturally occurring radioactive material present, especially the concentrations and quantities of nuclides such as actinium, lead, bismuth and thallium which are intermediate in the thorium series. External radiation levels from thorium nitrate in storage varies. A sampling indicates that one storage configuration, an isolated drum, yields contact exposure levels of approximately 5 mR/hr, one foot levels of approximately 2 mR/hr, and three foot levels of approximately 1 mR/hr. During vitrification chemical and physical reactions can result in changes in the relative concentrations and quantities of the naturally occurring radioactive materials as well as the non-radioactive constituents which provide attenuation of radiation. Further, piping and process vessels, product containers and waste/filtration systems will have various concentrations and quantities of naturally occurring radioactivity. The demonstration process will provide information regarding the distribution of naturally occurring radioactive material and resultant radiation fields associated with thorium nitrate vitrification.

AIRBORNE RADIOACTIVITY. Airborne radioactivity may occur during the vitrification process. Airborne radionuclides will include thorium decay series radionuclides as resuspended particulates and radon-220 gas. The concentrations may vary widely. There may be many situations, such as grinding and crushing, dry feed transfer, off-gassing, sampling and analytical procedures, maintenance, packaging, waste disposal and accidents that result in exposure to airborne radioactivity. Dust particles may contain alpha particle, beta particle and gamma photon emitting radionuclides which can irradiate internal organs of the body after inhalation or ingestion. The demonstration will provide information regarding the concentrations and control of airborne radioactivity associated with thorium nitrate vitrification.

SURFACE CONTAMINATION. Transferable surface contamination can be a source of inhalation and ingestion of radioactive materials. Therefore, control of surface contamination is an essential component of any radiological protection program. Operations that may result in contaminated surfaces include crushing and grinding, process equipment maintenance, agitation, and decontamination. The demonstration process will provide information regarding the control and monitoring of surface contamination associated with thorium nitrate vitrification.

Much of the above information is applied from industry recommendations:
NCRP (1993). National Council on Radiation Protection and Measurements. *Radiation Protection in the Mineral Extraction Industry*, NCRP Report No.118 (National Council on Radiation Protection and Measurements, Bethesda, Maryland).

DEGASSING STUDIES: SAFETY AND PROCESS DESIGN CRITERIA

Preliminary studies of surrogate nitrates (magnesium nitrate and calcium nitrate) have shown that heating rate and interaction with polyethylene can produce undesirable operating conditions. The following principal objectives and assessment methodologies are being considered to bracket

the safety, hazard, and process design issues for the de-nitration and dehydration of the thorium nitrate.

- Does the nitrate-organic mixture react violently?
- Demonstrate the safe operating window (compositional, thermal, temporal).
- Does the mixture 'froth' or expand upon treatment? Does it expand beyond its primary containment?
- Is the thoria (ThO_2) produced an airborne hazard? What is the size and extent of thoria dust upon treatment and handling?
- Determine off-gas characteristics (composition, volume, carry-over) as a function of process conditions (i.e. operating temperature, heat rate, etc.)

These criteria will be determined through a series of small scale evaluations of various mixtures of the drum components. From these studies the safe compositional and operating windows will be determined. Information pertaining to the off-gas composition and volume will also be obtained.

PPV STUDIES: PROCESS DESIGN CRITERIA

The following principal objectives and assessment methodologies are under consideration to provide process design and scale-up information for the PPV stage of the thorium nitrate processing.

- Determine the nature of the iron component in the system. How does the iron interact with the molten bath (metal or oxide)?
- What occurs during the heating of the drum and the residuals contained within (e.g. CaO ThO_2-Fe_xO_y)?
- Do detrimental or beneficial compounds form during processing? How can this formation be either minimized or optimized?
- What additives can be used to reduce the temperature and viscosity of the bath with minimal impact on final product volume or weight?

These criteria will be determined through both an evaluation of surrogate system in a plasma reactor and through a series of small scale evaluations of various mixtures of the drum components. From these studies the optimum compositional and operating windows will be

determined. Information pertaining to the PPV processing and process design for scale-up will be determined.

SURROGATE TESTING AT GA TECH

OVERVIEW. The Ga Tech test plan was developed to address two principle concerns for the PPV of the thoria drums: (1) off-gas composition and (2) the chemistry and segregation of the individual components in the final product.

The off-gas composition will be addressed through particulate collection and process gas monitoring. In addition, the process equipment will be wiped down post-processing. One of the most important safety/hazard assessments of the PPV stage is the control of carry-over of the very fine (potentially less than 1 micron) thoria particulates into the air pollution control (APC) system. Thermal denitration of thorium nitrate has consistently formed porous, low strength, low-density agglomerates of 20 to 30 micron size with much smaller ThO_2 crystallites (ORNL-2875, S.D. Clinton). Although the APC can remove the thoria particulates, the handling of radiological materials is governed by the philosophy of As-Low-As-Reasonably-Achievable (ALARA) and thus the PPV process must minimize any potential exposure scenarios associated with the operation and maintenance of the PPV unit. The need for adopting a first stage APC blow-back filter (with direct recycle to the melt chamber) will be assessed.

The second concern will be addressed through the analysis of the final product and the associated mass balance of the overall process. The materials used for surrogates will bracket a range of physical, chemical and thermodynamic properties of the thoria. One of the concerns with the bath chemistry is the ability to form mixed oxide phases (Ca-Fe-Mg(Th)-O). Since thoria is one of the most refractory oxides known (m.p. ~3250°C/3523°K), it would be beneficial to incorporate the thoria into a lower melting eutectic. The temperatures required to form dense thorium oxide was greatly reduced by small amounts of Al_2O_3 or CaO. $Th(NO_3)_4$ - $Al(NO_3)_3$ solutions to a flame calcination gave dense oxide spheres of 2.5 wt% Al_2O_3 at 2200°C (ORNL-3382, C.C. Haws). It is noteworthy that appreciable CaO is already contained in a number of the storage drum configurations. This will reduce the viscosity (ease of product removal), reduce the required bath (product) temperature, and reduce the vapor pressure of the thoria. This provides benefits from processing/availability, material of construction, and health/safety/maintenance perspectives.

GA TECH PLASMA APPLICATION RESEARCH FACILITY (PARF). The Ga Tech facility uses the 100KW plasma torch positioned over the center of a cone shaped graphite crucible in a primary reaction chamber. Feed is accomplished through a manual feed chute capable of handling 3" x 8.5" (7.62 cm x 21.6 cm) capsules. The crucible consists of a graphite block fabricated to form an internal cone 9-in (22.9 cm) deep with an upper cone diameter of 12-in (30.5 cm) and bottom cone diameter of 9-in. The primary chamber is connected via a hot gas sampling duct to the air pollution control (APC) system. The APC system consists of a quench/packed bed scrubber, water filled blower, bubbler tank, and associated sludge tank,

pumps, and recycle piping. The blower is capable of pulling several inches water column (W.C.) negative pressure on the primary chamber, and drives the APC flow through the bubbler and the stack.

The surrogate drum packages consist of metal (Fe) canisters filled with various amounts of the principle components of interest. The canisters will have their contents individually weighed and logged. The various surrogate canisters will be processed in three series consisting of a total of five different runs. The first series will consistent of three different runs with one run for each of the following two series. The feed rate will be between 5-10 minutes per canister depending upon the run configuration. It is expected that 5 to 15 pounds (2.27 to 6.8 Kg) of product will be formed per series.

SURROGATE FORMULATIONS. Since all isotopes of thorium are radioactive, a surrogate material has to be used to account for the chemical, physical, and thermodynamic properties of thoria for the Ga Tech testing. It was decided based upon literature values for the physical parameters and the thermal and chemical behavior of various materials to select two primary surrogate materials (MgO and ZrO_2) and one trace surrogate material (CeO_2). This will allow the various parameters to be bracketed for the thoria. Table III lists the various parameters for the three surrogate materials. It should be noted that Ce has been widely used as a surrogate for the uranium and plutonium systems. Thus, the partitioning and behavior data for the cerium will provide additional insight into the PPV of the major radiological elements of interests within the nuclear fuel cycle.

TABLE III. SURROGATE MATERIALS FOR ThO_2 PROCESSING

	ThO_2	MgO	ZrO_2	CeO_2
Formula Weight	264	40.3	123.2	172.1
Melting Point (°C)	3220±50	2852	2680	~2600
Boiling Point (°C)	4400	3600	4300	
Oxidation State	+4	+2	+4	+4 (+3)
Crystal Structure	cubic	cubic	monoclinic	cubic
Phase transformation	No	No	Yes (tetragonal, cubic)	Yes (hexagonal)

There will be five different configurations used to bracket the various drum configurations and to ensure that the off-gas and final product parameters can be obtained. Table IV lists the weight percent configurations to be used for the PPV evaluation of thoria processing.

TABLE IV
CANISTER COMPOSITIONS (WEIGHT%)
FOR SURROGATE PROCESSING

	Series #1			Series #2	Series #3
	Run #1	Run #2	Run #3	Run #1	Run #1
MgO	79.4 wt%	41.3 wt%	22.7 wt%	12.0 wt%	(1g)[A]
ZrO_2	(1g)[A]	(1g)[A]	(1g)[A]	(1g)[A]	12.0 wt%
CeO_2	(1g)[A]	(1g)[A]	(1g)[A]	(1g)[A]	(1g)[A]
CaO	0	24.0 wt%	24.0 wt%	27.4 wt%	27.4 wt%
C	0	0	18.6 wt%	21.1 wt%	21.1 wt%
Fe[B]	20.6 wt%	34.7 wt%	34.75 wt%	39.5 wt%	39.5 wt%
# canisters/run	9	9	12	27	27

A 1g of "tracer" surrogate is added to help bracket the surrogate performance. This amount is not calculated in the percent distribution of components.
B The Fe is present as the metal canister (i.e., drum surrogate).

SAMPLE COLLECTION AND ANALYSIS METHODS. The process parameters, the off-gas particulates, and the final product will be sampled. These measurements will provide input to safety, hazard and process design parameters for the pilot scale thorium PPV unit.

The process parameters will be measured in a fixed position in the hot duct (~800°C/1073°K) between the primary chamber and the air pollution control device. A turnkey CEM based sampling and analysis system will be used to determine: O_2, CO, CO_2, NO, NO_2, and THC. Additional data collected includes the gas velocity, static pressure, average moisture content, dry gas sampling rate and volume, and total sample gas flow.

The off-gas particulates will be collected in a fixed position in the hot duct using a United States Environmental Protection Agency (EPA) Method 5 sampling train with Teflon filter. The particulates will be analyzed using scanning electron microscopy and energy dispersive x-ray spectroscopy. The particle size, size distribution for the different elemental components, and the speciation of the particulates will be determined.

The final product will be removed at the end of each week after the reactor has been allowed to cool down. The bulk properties (mass, volume, bulk density, and visual appearance) along with the phase and elemental homogeneity of the final waste form will be determined. The

phases formed and the final disposition of the surrogate components will provide insight into the bath chemistry, the redox nature of the process, and the final disposition of the iron.

SUMMARY

The experiments planned for the evaluation of PPV technology for the processing of thorium surrogate materials at Ga Tech (PARF) should provide insight into the safety, hazard, and process design requirements in support of full scale operations. Although some of the variables from the low-temperature process are unknown, the high-temperature surrogate processing information will help support the low-temperature process development. Limited small quantity experiments using the actual thorium nitrate are necessary. The scope and methods to most efficiently obtain this data are currently being defined by the project team. For instance, benchmarking the controlled degas-mode heat-up rate and operating conditions with small quantities of the thorium nitrate feed matrix is required to fix the drum scale process design basis requirements. It is recognized that to obtain a relatively low bath temperature process (1550°C-2100°C/1823°K-2373°K), and low viscosity bath for flowability, other materials than those present (e.g., glass formers) may have to be added. It is possible that additions to the drum during low temperature processing may reduce the potential hazards associated with the low-temperature process and assist in the high-temperature process. Thus, the results from both series of processes and safety design criteria are being utilized in a synergistic manner to support the best overall design for the processing of the thorium nitrate crystals to safely produce a stable final product.

ACKNOWLEDGEMENTS

The authors would like to thank the Defense Logistics Agency for the opportunity to assist in developing the methodologies to assist in the reduction of materials currently in the Defense National Stockpile Center operations. A special thanks is given to Mr. Kevin Reilly of the DLA for his programmatic and administrative oversight and support of this program. The authors thank Mr. Paul Haas of the ORNL Chemical Technology Division for data supplied on thermal denitration of hydrated thorium nitrates, particulate characteristics and laboratory experience on the subject. The authors also thank Dr. Bill Bostick and Dr. Richard Hagenauer of the K-25 Technical Division for work related to developing a system for nondestructive assay (NDA) application to monitor thorium and its daughter activity throughout the air pollution control system. The authors also thank Mr. Craig Reed of the K-25 Health Physics Department for providing technical guidance associated with radiological hazards.

8-48-X17
#2

Georgia Tech
RESEARCH INSTITUTE

Georgia Tech Research Institute
Georgia Institute of Technology
Atlanta, Georgia 30332-0800
USA

February 15, 1996

Dr. Bud Camacho
President & CEO
Plasma Technology Corporation
6601 Six Forks Road Suite 400
Raleigh, NC 276

Dear Bud:

The quarterly process report (QPR) for November 1, 1995 to January 31, 1996 for the Plasma Processing of Surrogate Thorium Nitrate Crystals is attached. This QPR is delivery item #2. Please contact me for additional information as required.

Sincerely,

Robert (Bob) A. Newsom, Research Technologist II
Manager, Plasma Application Research Facility
Georgia Institute of Technology, Georgia Tech Research Institute
Electro-Optics, Environment, and Materials Laboratory
Environmental Engineering Branch
151 Sixth St. O'Keefe Bldg., Atlanta, GA. 30332-0837
404-894-8047, Fax 404-894-2184

QUARTERLY PROGRESS REPORT

Deliverable:	**#2**
Title:	**Plasma Processing of Surrogate Thorium Nitrate Crystals** **PTC Contract for Prime :#DE-AC05-840R21440**
Department(s):	Georgia Institute of Technology
Main #:	D-48-X17: Architecture Construction Research Center
Sub(s) #:	A-5017-000 Georgia Tech Research Institute
Contract Duration:	12 months: July 24, 1995 to July 23, 1996
Reporting Period:	**November 1, 1995 to January 31, 1996**
Date Due:	February 15, 1996
Prepared by:	Robert A. Newsom

Introduction

Plasma Technology is being developed at the Georgia Tech Plasma Application Research Facility (PARF). Various programs are currently being investigated for the improved operation and processing of plasma applications. The programs consist of using the plasma torch for research on asbestos destruction, municipal waste processing, incinerator ash processing, soil remediation, soil stabilization, and surrogate testing of both hazardous and low-level radioactive wastes. Plasma Technology Corporation (PTC) is developing system designs and implementations for processing materials for various organizations including the Defense Logistics Agency (DLA). The DLA materials requiring treatment includes thorium nitrate crystals.

The Plasma Application Research Facility at Georgia Institute of Technology will provide testing and engineering support for the development activities by Plasma Technology Corporation for these efforts. The program participants at Georgia Tech will consist of the Construction Research Center (GT/CRC) and the Georgia Tech Research Institute (GTRI).

Project Summary & Goals

The initial tasks for this program will consist of the treatment of thorium nitrate crystals. A test plan is to be generated, procurement of materials will be conducted, a system design criteria will be established, and the conduction of tests will be performed. The conduction of tests includes data collection, sample collection, data analysis, and reports. The program will use a team approach with collaboration between the government, industry, PTC and Georgia Tech staff members. The program will advance the processing and development of plasma technology for degassing and vitrification of materials specifically thorium nitrate crystals A surrogate of thorium nitrate will be used at the Georgia Institute of Technology Plasma Application Research Facility.

Report Period Accomplishments:

A meeting to discuss the results of the Ga Tech Test phase of this program was held at the Oak Ridge facility on November 11, 1995

Project Meeting Attendees: 10:30AM to 4:30PM

Name	Organization	Phone
Kevin Reilly	DLA	
Frank Van Ryn	LMES	615-574-1907
Bill Hermes	LMES	651-574-9568
Bob Newsom	Georgia Tech	404-894-8047
Luther Gibson	LMES	
Hom-Ti Lee	K-25	615-574-1603

The purpose of this meeting was to discuss the surrogate test program at Ga Tech and the overall program results for processing the Thorium Nitrate materials for disposal by DLA during this test phase.

Bill Hermes gave an overview of the surrogate test results review process and data summary. Luther Gibson presented the continuous emissions monitoring (CEM) background. Bob Newsom addressed the Ga Tech close-out issues. Bill Hermes discussed the product chemistry technology based on the data and PPV design requirements. The process history design basis for the lab pilot scale demonstration scale was presented by Bill Hermes.

A logical flow diagram for the decision criteria was presented and discussed by the group. A chemistry lab scale test and analysis of the material is planned before a final decision is reached. LMES will perform this test. The pilot scale demonstration decision will be determined after the chemistry lab test is performed.

Next Period Plans: February 1, 1996 to May 31, 1996

1. The clean-up of the Ga Tech test will be completed and the task is completed.

2. LMES with Ga Tech input will assemble the test report.

Financial Report

For this quarterly reporting period charges on the GTRI task (A5017) are $15300.83. A balance of $798.26 remains on the GTRI task account A5017 at the end of the period. The Construction Research Center (CRC) main project account D-48-X17 for this period had charges of $18251.70 leaving a balance of $-2,139.45 (negative) remaining on the CRC account. A total program balance (GTRI/A5017 + CRC/D-48-X17) is $-1,431.19 on the program account for at the end of this quarterly period.

0-48-X17
#3

Georgia Tech Research Institute

May 15, 1996

Dr. Bud Camacho
President & CEO
Plasma Technology Corporation
6601 Six Forks Road Suite 400
Raleigh, NC 27615

Dear Bud:

The quarterly process report (QPR) for February 1, 1996 to April 30, 1996 for the Plasma Processing of Surrogate Thorium Nitrate Crystals is attached. This QPR is delivery item #3. Please contact me for additional information as required.

Sincerely,

Robert (Bob) A. Newsom, Research Technologist II
Manager, Plasma Application Research Facility
Georgia Institute of Technology, Georgia Tech Research Institute
Electro-Optics, Environment, and Materials Laboratory
Environmental Engineering Branch
151 Sixth St. O'Keefe Bldg., Atlanta, GA. 30332-0837
404-894-8047, Fax 404-894-2184

QUARTERLY PROGRESS REPORT

Deliverable:	**#3**
Title:	**Plasma Processing of Surrogate Thorium Nitrate Crystals PTC Contract for Prime :#DE-AC05-840R21440**
Department(s):	Georgia Institute of Technology
Main #:	D-48-X17: Architecture Construction Research Center
Sub(s) #:	A-5017-000 Georgia Tech Research Institute
Contract Duration:	12 months: July 24, 1995 to July 23, 1996
Reporting Period:	**February 1, 1996 to April 30, 1996**
Date Due:	May 15, 1996
Prepared by:	Robert A. Newsom

Introduction

Plasma Technology is being developed at the Georgia Tech Plasma Application Research Facility (PARF). Various programs are currently being investigated for the improved operation and processing of plasma applications. The programs consist of using the plasma torch for research on asbestos destruction, municipal waste processing, incinerator ash processing, soil remediation, soil stabilization, and surrogate testing of both hazardous and low-level radioactive wastes. Plasma Technology Corporation (PTC) is developing system designs and implementations for processing materials for various organizations including the Defense Logistics Agency (DLA). The DLA materials requiring treatment includes thorium nitrate crystals.

The Plasma Application Research Facility at Georgia Institute of Technology will provide testing and engineering support for the development activities by Plasma Technology Corporation for these efforts. The program participants at Georgia Tech will consist of the Construction Research Center (GT/CRC) and the Georgia Tech Research Institute (GTRI).

Project Summary & Goals

The initial tasks for this program will consist of the treatment of thorium nitrate crystals. A test plan is to be generated, procurement of materials will be conducted, a system design criteria will be established, and the conduction of tests will be performed. The conduction of tests includes data collection, sample collection, data analysis, and reports. The program will use a team approach with collaboration between the government, industry, PTC and Georgia Tech staff members. The program will advance the processing and development of plasma technology for degassing and vitrification of materials specifically thorium nitrate crystals A surrogate of thorium nitrate will be used at the Georgia Institute of Technology Plasma Application Research Facility.

Report Period Accomplishments:

During this period input to the final report was be provided to LMES for their preparation of the report including Georgia Tech Test phase.

Next Period Plans: May 1, 1996 to July 23, 1996

The Final Report will be prepared and submitted by Georgia Tech and LMES.

Georgia Tech Research Institute

D-48-X17
#4

July 23, 1996

Dr. Bud Camacho
President & CEO
Plasma Technology Corporation
6601 Six Forks Road Suite 400
Raleigh, NC 27615

Dear Bud:

The Final Report for the Plasma Processing of Surrogate Thorium Nitrate Crystals is attached. This Final Report is delivery item #4. Please contact me for additional information as required.

Sincerely,

Robert (Bob) A. Newsom, Research Technologist II
Manager, Plasma Application Research Facility
Georgia Institute of Technology, Georgia Tech Research Institute
Electro-Optics, Environment, and Materials Laboratory
Environmental Engineering Branch
151 Sixth St. O'Keefe Bldg., Atlanta, GA. 30332-0837
404-894-8047, Fax 404-894-2184

FINAL REPORT

Deliverable:	**#4**
Title:	**Plasma Processing of Surrogate Thorium Nitrate Crystals PTC Contract for Prime :#DE-AC05-840R21440**
Department(s):	Georgia Institute of Technology
Main #:	D-48-X17: Architecture Construction Research Center
Sub(s) #:	A-5017-000 Georgia Tech Research Institute
Contract Duration:	12 months: July 24, 1995 to July 23, 1996
Reporting Period:	**July 24, 1995 to July 23, 1996**
Date Due:	July 23, 1996
Prepared by:	Robert A. Newsom and LMES

Introduction

Plasma Technology is being developed at the Georgia Tech Plasma Application Research Facility (PARF). Various programs are currently being investigated for the improved operation and processing of plasma applications. The programs consist of using the plasma torch for research on asbestos destruction, municipal waste processing, incinerator ash processing, soil remediation, soil stabilization, and surrogate testing of both hazardous and low-level radioactive wastes. Plasma Technology Corporation (PTC) is developing system designs and implementations for processing materials for various organizations including the Defense Logistics Agency (DLA). The DLA materials requiring treatment includes thorium nitrate crystals.

The Plasma Application Research Facility at Georgia Institute of Technology will provide testing and engineering support for the development activities by Plasma Technology Corporation for these efforts. The program participants at Georgia Tech will consist of the Construction Research Center (GT/CRC) and the Georgia Tech Research Institute (GTRI).

Project Summary & Goals

The initial tasks for this program will consist of the treatment of thorium nitrate crystals. A test plan is to be generated, procurement of materials will be conducted, a system design criteria will be established, and the conduction of tests will be performed. The conduction of tests includes data collection, sample collection, data analysis, and reports. The program will use a team approach with collaboration between the government, industry, PTC and Georgia Tech staff members. The program will advance the processing and development of plasma technology for degassing and vitrification of materials specifically thorium nitrate crystals A surrogate of thorium nitrate will be used at the Georgia Institute of Technology Plasma Application Research Facility.

K-25

K/EM-409

OAK RIDGE
K-25 SITE

LOCKHEED MARTIN

DEFENSE LOGISTICS AGENCY

THORIUM NITRATE SURROGATE

TEST RESULTS REPORT

FEBRUARY 1996

Pepared by
the Oak Ridge K-25 Site
Oak Ridge, Tennessee 37831-7133
managed by
LOCKHEED MARTIN ENERGY SYSTEMS, INC.
for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-84OR21400

MANAGED BY
LOCKHEED MARTIN ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

UCN-13671 (16 6-95)

K/EM-409

DEFENSE LOGISTICS AGENCY

THORIUM NITRATE SURROGATE

TEST RESULTS REPORT

W. H. Hermes, Central Engineering Services
D. P. Hoffmann, Technical Support Organization
L. V. Gibson, Analytical Services Organization
L. A. Harris, Technical Support Organization
H. T. Lee, Technical Support Organization
F. Van Ryn, Environmental Management Division

February 1996

Work sponsored by the Defense Logistics Agency, Department of Defense, under Interagency Agreement 1872-H070-A1 with the U.S. Department of Energy (DOE) and performed by Lockheed Martin Energy Systems, Inc., under DOE Contract DE-AC05-84OR21400.

Prepared by
the Oak Ridge K-25 Site
Oak Ridge, Tennessee 37831-7133
managed by
LOCKHEED MARTIN ENERGY SYSTEMS, INC.
for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-84OR21400

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ACRONYMS AND ABBREVIATIONS

A	amps
ALARA	As-Low-As-Reasonably-Achievable
Al_2O_3	aluminum oxide (alumina)
APC	Air Pollution Control
BEI	backscattered electron image
Ca	Calcium
CaO	Calcium oxide (calcia)
Ce	Cerium
CEM	Continuous Emission Monitor
CeO_2	Cerium oxide (ceria)
CO	Carbon Monoxide
CO_2	Carbon Dioxide
DLA	Defense Logistics Agency
DNSC	Defense National Stockpile Center
DOE	U.S. Department of Energy
DOF	degree(s) of freedom
dscf	dry standard cubic feet
dscfh	dry standard cubic feet per hour
EDS	energy-dispersive spectroscopy
EPA	U.S. Environmental Protection Agency
Fe	iron
FID	Flame ionization detector
Georgia Tech	Georgia Institute of Technology
Mg	Magnesium
MgO	magnesium oxide (magnesia)
min	minute
NCRP	National Council on Radiation Protection and Measurements
NDA	Nondestructive Analysis
NDIR	Nondispersive infrared
NO_2	Nitrogen Dioxide
NO	Nitrogen Oxide
N-T	Non-Transferred
O_2	Oxygen
PARF	Plasma Application Research Facility
PPV	Plasma Pyrolysis Vitrification
Rx	Reaction
scfm	standard cubic feet per minute
SEM	scanning electron microscopy
TGA	Thermogravemetric
^{232}Th	Thorium
THC	Total Hydrocarbons
ThO_2	Thorium Oxide (thoria)
TN	Thorium Nitrate
^{233}U	Uranium-233

ACKNOWLEDGEMENTS

The authors wish to acknowledge several individuals who made significant contributions to the implementation of this project. F. Kevin Reilly, the Defense Logistics Agency's Program Manager for the project, was an invaluable resource throughout the project. The project's technical design, planning, and field execution were accomplished through the efforts of Dr. S. L. Camacho (Plasma Technology Corp.), Dr. Louis J. Circeo, Lamar Carney, Robert A. Newsome, and Geoving Gerard (Georgia Institute of Technology). Dr. Paul Haas (Oak Ridge National Laboratory) conducted the thorium nitrate research that laid the groundwork for this study. Dr. William Bostick (Lockheed Martin Energy Systems) provided invaluable support in the selection of surrogate materials used to test the vitrification process. The hot gas sampling and continuous emissions monitoring (CEM) support team consisted of Bob Gross, Mark Burdette, and Allen Blevins of Energy Systems Analytical Services Organization. Craig Reed (K-25 Site Health Physics Department) provided health physics guidance for the project. Jimmy Allen of Clean Air Engineering supplied and set up the CEM equipment. Digital imaging support was provided by Daniel Phifer and Joe Stone; information management and publishing support, by Doris Dyer, Nancy Smith, and Joe Stone (Energy Systems).

EXECUTIVE SUMMARY

Thorium (^{232}Th) can be utilized as fuel in breeder style nuclear reactors. ^{232}Th is considered a fertile nuclide since it can be fissioned by high energy (>1 MeV) neutrons to produce fissile uranium (^{233}U). The Defense National Stockpile Center (DNSC) currently has ^{232}Th in storage in the chemical form of thorium nitrate, $Th(NO_3)_4 \cdot 4H_2O$. The potential use of ^{232}Th is being phased out; hence, there is a need to mitigate any potential future environmental or exposure hazards associated with this material. Since ^{232}Th has a long half-life (1.41×10^{10} years), the current program is evaluating treatment methodologies to safely produce the less leachable oxide. The more chemically stable oxide is lower in mass and volume and exhibits low leachability characteristics, making it suitable for licensed long-term storage.

The Defense Logistics Agency (DLA) has approximately 4500 metric tons of thorium nitrate (includes drummed constituents) and has sought assistance from the U.S. Department of Energy (DOE) in evaluating plasma torch technology, based on DOE's experience in handling and treating radioactive material. This report presents the results from surrogate testing experiments using the Georgia Institute of Technology (Georgia Tech) Plasma Application Research Facility (PARF). The experiments at Georgia Tech were conducted in September 1995 using nonradiological surrogates for the thorium oxide in combination with typical proportions of drummed constituents, using a mole-percent oxide basis. Without adding bath modifiers (to reduce temperature), whole drum melting was simulated using iron feed canisters to determine the fate of the iron from the drums.

General characteristics of the vitrified product, off-gas chemical analysis, and particulate carry-over data were obtained. The testing was videotaped and photographed. Feed data and operating conditions (including variations) were documented. Important processing conclusions from the surrogate testing are as follows.

- Modifier must be added to achieve a lower melt temperature.
- Iron should be removed prior to vitrification (i.e., remove the outer metal drums).
- Asphalt and aluminum oxide bricks need to be removed (from the Indiana inventory) prior to treatment.

Figure ES.1, a Logic Diagram, identifies the information necessary to determine the feasibility of using plasma torch heating in the treatment scheme for the thorium nitrate. The logic diagram illustrates the need to determine degassing properties of the thorium nitrate and bath characteristics of the melt. The cost centers, as indicated in Figure ES.1, are pretreatment, thermal treatment and associated processes, and residual shipment and acceptance.

Data from lab-scale degassing testing will be evaluated before proceeding to characterize bath chemistry. Section 3 provides an overview of the types of experiments using actual thorium nitrate that are needed to evaluate the application of plasma processing to DLA's thorium nitrate inventory goals. If overall lab- and bench-scale testing validate plasma torch heating technology as viable, in conjunction with acceptance criteria of residuals at western repositories, a pilot-scale demonstration will be conducted at the K-25 Site in Oak Ridge, Tennessee.

1. INTRODUCTION

1.1 SURROGATE TESTING AT GEORGIA TECH

To determine the feasibility of using plasma to vitrify the Defense Logistics Agency's (DLA's) thorium nitrate, a test plan (Hermes et al. 1995) was developed that would address three major concerns for the Plasma Pyrolysis Vitrification (PPV):

- the chemistry and segregation of the individual components in the final product;
- the fate of iron and the material balance baseline; and
- the off-gas composition/mass carry-over.

To take advantage of plasma heating's capacity to generate high temperature and minimize pretreatment costs, whole, unopened DLA drums were simulated. The surrogate testing was based on thoria, versus thorium nitrate; therefore, a low-temperature degassing process was needed to convert to the oxide prior to plasma treatment. [Surrogate oxides were used to represent the thoria (ThO_2).] An overview of the DLA Thorium Nitrate Plasma Program is provided in Appendix A.

The product chemistry was addressed through the chemical analysis of the final products. An overall mass balance was completed. The materials used for surrogates bracketed a range of physical, chemical, and thermodynamic properties of the thoria. A bath chemistry concern is the ability to form mixed oxide phases (Ca-Fe-Mg(Th)-O). Since thoria is the most refractory oxide known (melting point approximately 3250°C/3523°K) (Ryshkewitch and Richerson 1985), it is beneficial to incorporate the thoria into a lower melting eutectic. Based on previous processing, temperatures required to form dense thorium oxide are greatly reduced by small amounts of Al_2O_3 or CaO. Solutions of $Th(NO_3)_4$—$Al(NO_3)_3$ subjected to flame calcination gave dense oxide spheres of 2.5 wt % Al_2O_3 at 2200°C (Haws and Haas 1963). It is noteworthy that appreciable CaO is already contained in a number of the storage drum configurations. This offers the potential to reduce the viscosity (ease of product removal), reduce the required bath (product) temperature, and reduce the vapor pressure of the thoria. This provides benefits from the perspectives of processing/availability, material of construction, and health/safety/ maintenance.

The off-gas characterization was addressed through hot zone process gas composition monitoring and particulate collection (carry-over). The process equipment was cleaned out after processing. An important safety/hazard assessment of the PPV is the control of carry-over of the very fine (potentially less than 1 micron) thoria particulates into the air pollution control (APC) system. Thermal denitration of thorium nitrate has consistently formed porous, low strength, low-density agglomerates of 20–30 micron size with much smaller thorium oxide (ThO_2) crystallites (Haws and Haas 1963). The APC can remove the thoria particulates, but the handling of radiological materials is governed by the as-low-as-reasonably-achievable (ALARA) philosophy; thus, the PPV process must minimize any potential exposure scenarios associated with the operation and maintenance of the PPV unit.

Figure 1.1. Photographs of the Georgia Tech plasma torch.

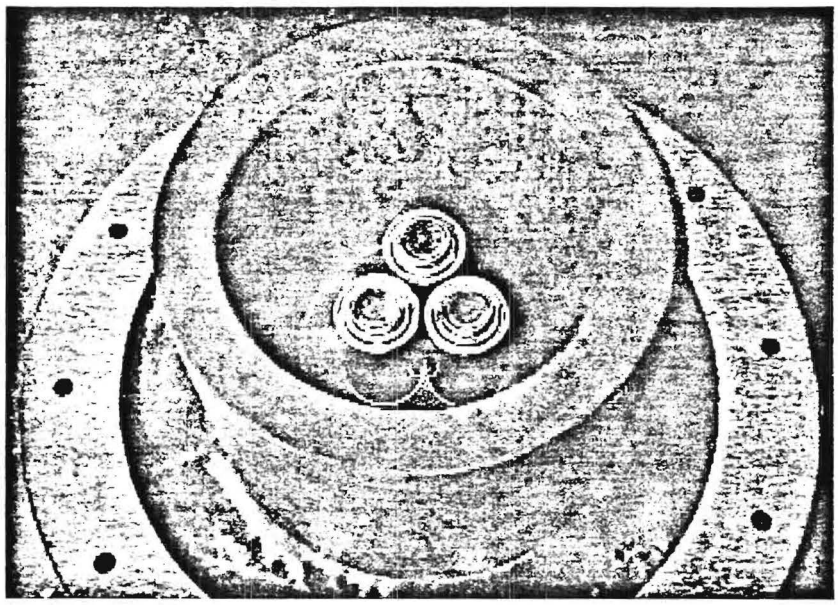


Figure 1.2. The graphite crucible with surrogate canisters prior to processing.

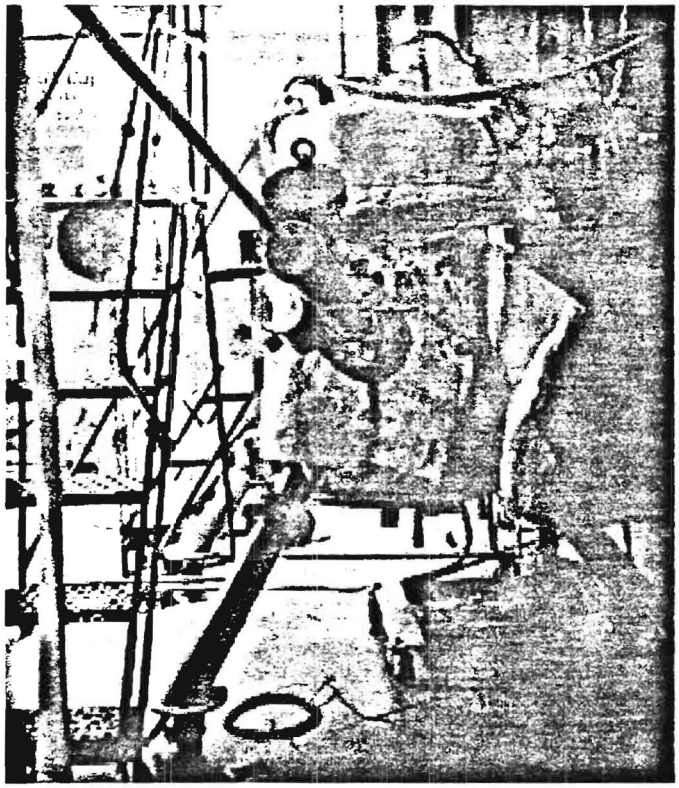


Figure 1.3. The Georgia Tech plasma reactor during operation.

Table 1.1. Surrogate materials for thorium oxide processing

	ThO_2	MgO	ZrO_2	CeO_2
Formula Weight	264	40.3	123.2	172.1
Melting Point (°C)	3220±50	2852	2680	~2600
Boiling Point (°C)	4400	3600	4300	
Oxidation State	+4	+2	+4	+4 (+3)
Crystal Structure	cubic	cubic	monoclinic	cubic
Phase transformation	No	No	Yes (tetragonal, cubic)	Yes (hexagonal)

Table 1.2. Canister compositions (weight percent) for surrogate processing

	Series 1			Series 2
	Run 1	Run 2	Run 3	Run 1
MgO	71.3 wt %	41.3 wt %	22.7 wt %	0
ZrO_2	(1g)[a]	(1g)[a]	(1g)[a]	29.5 wt %
CeO_2	(1g)[a]	(1g)[a]	(1g)[a]	(1g)[a]
CaO	0	24.0 wt %	24.0 wt %	22.0 wt %
C	0	0	18.6 wt %	17.0 wt %
Fe[b]	28.7 wt %	34.7 wt %	34.70 wt %	31.5 wt %
Number of canisters fed per run	14	9	12	18

[a]1g of "tracer" surrogate is added to help bracket the volatility performance. This amount is not calculated in the percent distribution of components.

[b]The iron is present as the metal canister (i.e., drum surrogate).

1.4 SAMPLE COLLECTION AND ANALYSIS METHODS

Process parameters were measured simultaneously with hot zone off-gas composition, and hot zone particulate collection. These measurements provide input to the safety, hazard, and process design parameters for the PPV unit.

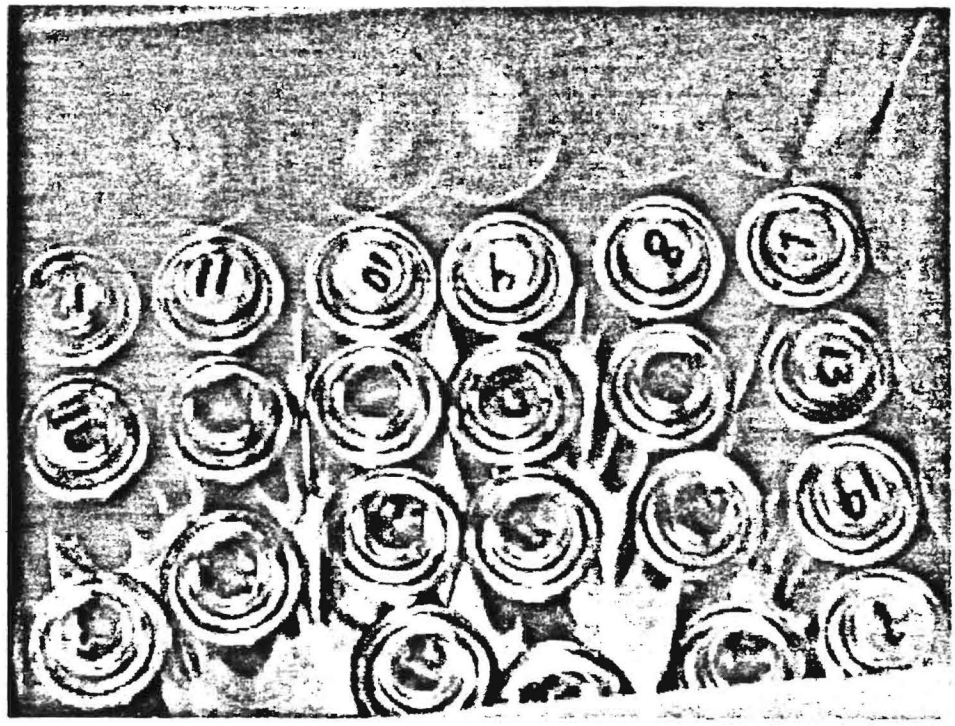


Figure 1.4. Photograph of canisters used to simulate the metal drums in which the thorium nitrate is stored.

The off-gas parameters were measured in a fixed position in the hot duct between the primary chamber and the air pollution control device. Two photographs of the 4-foot hot gas sampling spool piece are included as Figure 1.5. A continuous emission monitor (CEM) based sampling and analysis system was used to determine O_2, CO, CO_2, NO, NO_2, and THC. The CEM sampling point was between the primary chamber and the spool piece. Additional data collected includes the gas velocity, static pressure, average moisture content, dry gas sampling rate and volume, and total sample gas flow. A photograph of the CEM system components (trailer mounted) is included as Figure 1.6.

The off-gas particulates were collected in a fixed position in the hot duct using a U.S. Environmental Protection Agency (EPA) Method 5 sampling train with Teflon filter. The particulates were analyzed using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). The composition of the particulate matter was determined; data for particle size/speciation (composition per particle) were considered not to be necessary.

After reactor cooldown, the final product was removed at documented points in the system following each of the two continuous tests. The exact location and properties (mass, volume, bulk density, and visual appearance) along with phase and elemental homogeneity of the final waste form were determined. The phases formed and the final disposition of the surrogate components provide insight into the bath chemistry and the final disposition of the iron.

1.5 SPECIFIC TEST OBJECTIVES

The key test objectives as documented in the test plan are the following.

1. Conduct two to four tests.
2. Gain pilot scale experience for using oxide surrogates.
3. Baseline thermal NO_x for air generated plasma.
4. Collect off-gas sampling data for CO, CO_2, NO, NO_2, O_2 and THC.
5. Quantify particulate carry-over and general volatility trends.
6. Ascertain practicality of product matrix formation (i.e., density, leachability) with insight gained into modifier requirements at pilot scale.
7. Quantify the fate and form of iron and its impact on pretreatment/pilot scale design.
8. Obtain specific materials of construction experience with graphite crucible.
9. Obtained data for thermal profile and off-gas flow variance with time.
10. Complete unitized iron packaged feed simulating DLA drums directly to iron melt surface, adjacent to nontransferred (N-T) plasma torch.
11. Identify important process control, operating, and pilot demo scale design parameters.
12. Bound the scope of further lab, bench, and pilot scale testing to accomplish the overall project.

All objectives were completed and results are documented herein.

1.6 CONCLUSIONS

Specific technical conclusions can be found in each subsection of the report. Presented in this section is an overall methodology for applying plasma process technology to the thorium nitrate inventory.

A Logic Diagram (see Figure ES.1) was developed to aid in understanding the key technical decisions related to managing the thorium nitrate inventory. This diagram is based on the need for degassing the thorium nitrate in a low-temperature furnace, separate from the PPV reactor. The highest technology development design complexity is tied to whole-drum processing, but whole-drum processing yields the lowest front-end handling and pretreatment costs. The Georgia Tech surrogate testing was premised on whole drum processing because plasma heating is the only available technology with the capacity to vitrify the whole drum.

The surrogate testing was done in part to evaluate the fate of iron, and the results demonstrate that iron is problematic in several ways and should be removed prior to plasma heating. This conclusion, based on completed bench-scale testing, directs attention to the second column which deals with a lower initial total feed stream but with the added burden of handling/removing the iron outer drum from each packaged unit. The remaining mass constituents are thorium nitrate, $Ca(OH)_2$, polyethylene, as defined in Appendix A. Since the removal of the iron is required, the Indiana drum (see Section 3.1—~825 lb per drum) will be reduced by the asphalt, Al_2O_3, and both the outer and inner iron drums, thus leaving the thorium nitrate, $Ca(OH)_2$ and polyethylene bags/fiber drum. To best promote the decision to deploy the PPV system based on ability to form a melt, eliminating the asphalt makes good design sense because it avoids evaluating the safety concern associated with a separate furnace.

Laboratory-scale testing, using gram quantities, is required to assess whether the thorium nitrate can be degassed safely in the presence of these hydrocarbons (polyethylene/fiber ~CH_2). The reactions between nitrates and hydrocarbons can be very exothermic with immediate temperature spikes and frothing within the containment structure. Based on safe degassing (calcination to 600°C) of the thorium nitrate to ThO_2, $Ca(OH)_2$ to CaO and CH_2 (polyethylene/fiber) to C, the next step will be to assess the degree to which a flowable melt can be formed. The CaO constituent in the drum will help reduce the melt temperature to something lower than the melting point of ThO_2, but based on the Georgia Tech surrogate testing, a controllable melt will not form without the addition of flux materials (i.e., fusion promoters). The target bath operating temperature for a PPV system would be a nominal 1700°C per good design practice, in order to reduce material failure.

To determine the design and material requirements for the PPV pilot system, verification of melt formation chemistry at this temperature is required. A simple, small-scale electric furnace can be used to complete melt chemistry definition. The decision to proceed to lab-scale melt chemistry evaluation is based on the success of the lab-scale gram quantity degas testing and the projected need for a vitrified product to meet repository acceptance requirements.

It is noteworthy that fusion promoters and secondary waste estimates are not reflected in the gross mass balance estimate presented in Figure ES.1. These are issues that are recognized to have significant impact on the overall inventory management program cost. Based on the current information, the Logic Diagram displays the need for pretreatment cost penalties to be defined to the extent of estimating costs for full-scale iron/drum handling and removal. The need for lab-scale testing is tied to the safety of the degas furnace operation and will be completed to evaluate to what extent separation of polyethylene (and organic fiber) is required. Residual management cost considerations are not addressed herein, but calculations were done to bracket the PPV process residuals with and without the iron. The basis used was one MD-1 drum (predominant storage unit configuration), and the level of radioactivity was calculated as a function of calcined drum constituent loading and density. The results, with exact basis, are presented in Appendix F. Secondary waste was ignored but is a significant issue with melter technologies.

The shipment volume per drum, based on PPV processing, can vary 10:1 based on constituent loading, and then that volume vary 2:1 based on the density range selected. For a nominal density of 3 g/cc and 20% constituent loading (per glass industry analog), the product volume per MD-1 drum is 5.28 ft^3. This yields an activity of 9.39 in Ci/g product and activity per unit volume equivalent to 5.32×10^5 nCi/ft^3, where the product material is assumed to occupy the volume at 3 g/cc, plus 50% more to represent a container (called "overpack" in Appendix F). This is consistent with the Nevada Test Site acceptance volume basis. It is clear that a vitrification technology development/deployment pathway could be enhanced by evaluating the use of "waste" materials that can function as bath fusion promoters (i.e., asbestos materials), should an economy of scale for vitrification be justified.

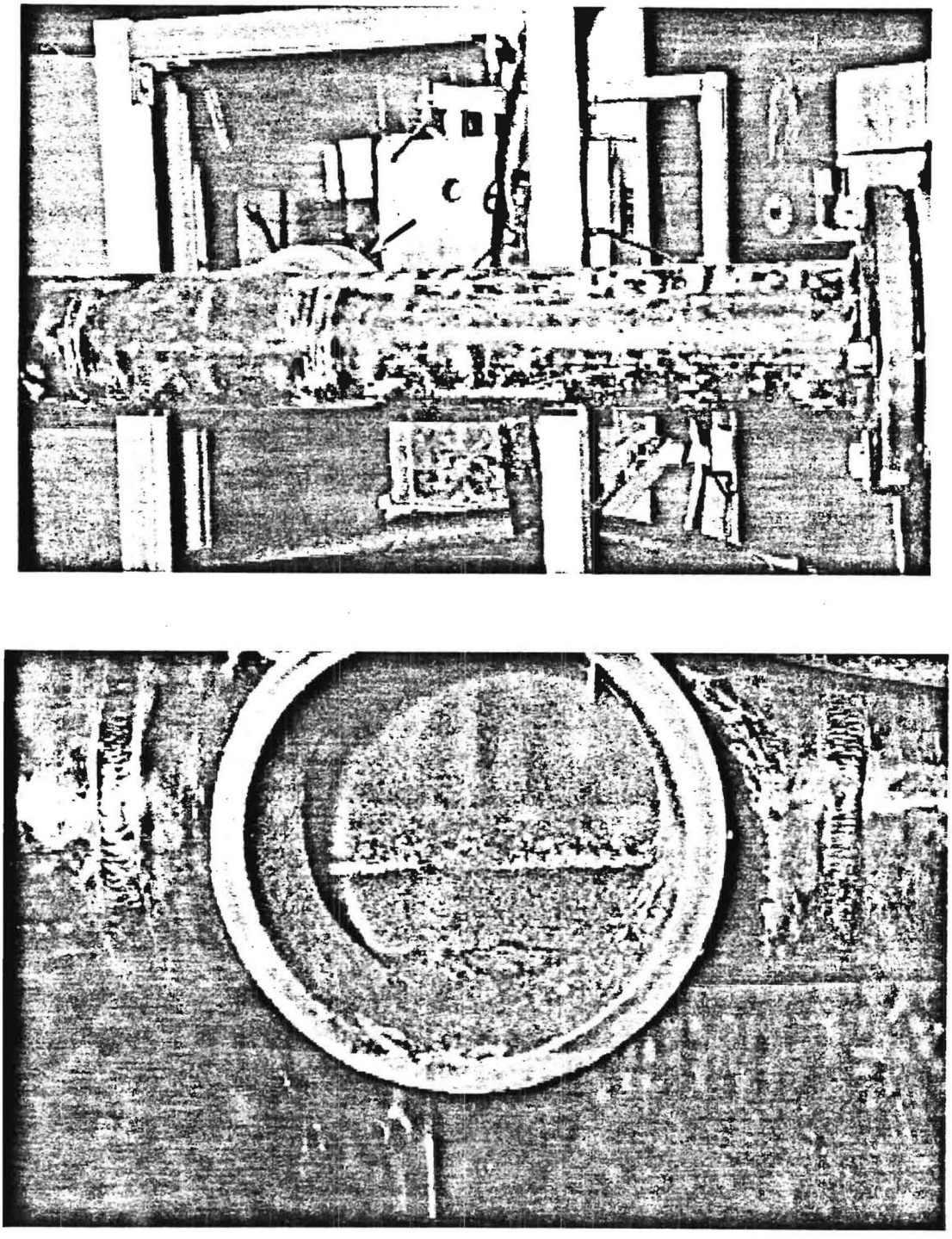


Figure 1.5. Hot gas sampling port. (Bottom photo shows the particulate byproduct of the vitrification process.)

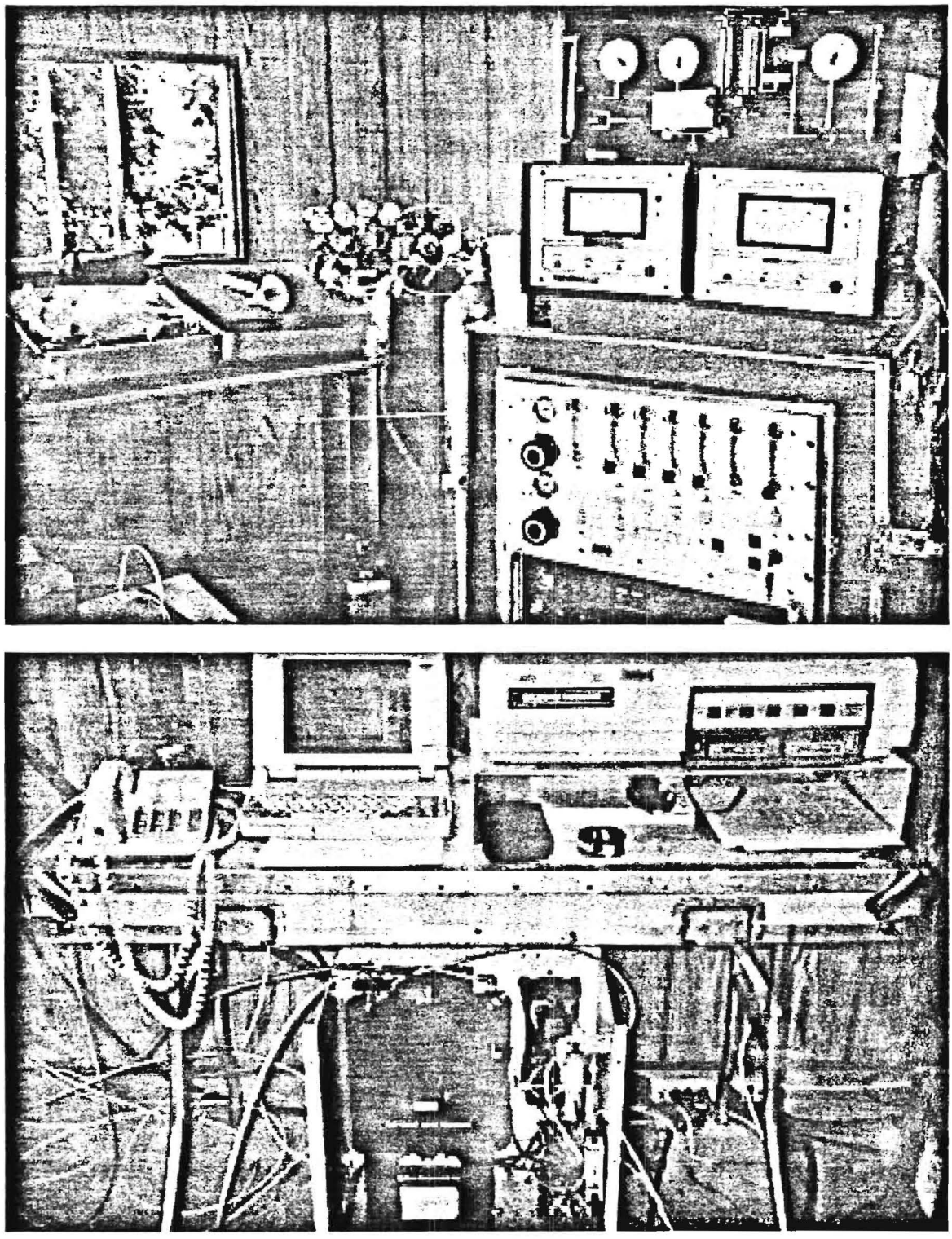


Figure 1.6. Continuous emission monitoring (CEM) system components.

2. TEST RESULTS

2.1 DESCRIPTION OF TEST EXECUTION AND PROCESS SUMMARY

Two series of surrogate feed tests were completed. Series 1 testing was completed September 11–12, 1995, and was comprised of the following segments: Hot Shakedown; Preheat; Series 1 (Run 1 Surrogate); Series 1 (Run 2 Surrogate); and Series 1 (Run 3 Surrogate). The surrogate used for ThO_2 for Series 1 was MgO. The feed materials summary data (introduced during the Preheat and Series 1 testing) is provided as Table 2.1.

Series 2 testing was completed September 19, 1995, and was composed of the following segments: Preheat and Series 2 (surrogate). The surrogate used for ThO_2 for Series 2 was ZrO_2. The feed materials summary data (introduced during the Preheat and Series 2 testing) is provided as Table 2.2.

The timing for introducing surrogate feed cans for Series 1 and 2 are presented in Tables 2.3 and 2.4, respectively (the exact compositional data, per surrogate feed can, is provided in Appendix C).

Table 2.1. Plasma testing—Series 1

	Weight distribution of feed materials (grams)					
Test Segment	Fe	MgO	CaO	C	ZrO_2	CeO_2
Preheat	7,090	0	0	0	0	
Run 1	1,120	2,818	0	0	14	14
Run 2	720	866	505	0	9	9
Run 3	960	635	670	520	12	12
Subtotal	9,890	4,319	1,175	520	37	37
% Total (15,978 g)	61.9	27.0	7.4	3.3	0.2	0.2

Table 2.2. Plasma testing—Series 2

	Weight distribution of feed materials (grams)[a]					
Test Segment	Fe	MgO	CaO	C	ZrO_2	CeO_2
Precharge (cold)	4,538	0	0	0	0	
Preheat (hot)	14,158	0	0	0	0	
Run 1	1,462	0	1,017	786	1,361	18
Subtotal	20,158	0	1,017	786	1,361	18
% Total (23,340 g)	86.4	0	4.4	3.4	5.8	0.1

[a]The last canister partially melted in the feed tube; 249 g of powder was collected.

Table 2.3. Series 1, surrogate addition[a]

Test segment	Feed event	Time	Elapsed time (min.)[b]
Series 1, Run 1	Can 1	12:12	
Series 1, Run 1	Can 2	12:18	6
Series 1, Run 1	Can 3	12:24	6
Series 1, Run 1	Can 4	12:31	7
Series 1, Run 1	Can 5	12:34	3
Series 1, Run 1	Can 6	12:40	6
Series 1, Run 1	Can 7	12:43	3
Series 1, Run 1	Can 8	12:47	4
Series 1, Run 1	Can 9	12:50	3
Series 1, Run 1	Can 10	12:54	4
Series 1, Run 1	Can 11	12:59	5
Series 1, Run 1	Can 12	13:06	7
Series 1, Run 1	Can 13	13:13	7
Series 1, Run 1	Can 14	13:19	6
Series 1, Run 2	Can 1	13:55	
Series 1, Run 2	Can 2	14:01	6
Series 1, Run 2	Can 3	14:07	6
Series 1, Run 2	Can 4	14:12	5
Series 1, Run 2	Can 5	14:18	6
Series 1, Run 2	Can 6	14:21	3
Series 1, Run 2	Can 7	14:28	7
Series 1, Run 2	Can 8	14:32	4
Series 1, Run 2	Can 9	14:35	3
Series 1, Run 3	Can 1	14:47	
Series 1, Run 3	Can 2	14:49	2
Series 1, Run 3	Can 3	14:52	3
Series 1, Run 3	Can 4	14:55	3
Series 1, Run 3	Can 5	14:57	2
Series 1, Run 3	Can 6	15:01	4
Series 1, Run 3	Can 7	15:03	2
Series 1, Run 3	Can 8	15:05	2
Series 1, Run 3	Can 9	15:08	3
Series 1, Run 3	Can 10	15:10	2
Series 1, Run 3	Can 11	15:12	2
Series 1, Run 3	Can 12	15:13	1

[a]The official record of material feed and process data for all Series 1 and Series 2 test segments, as well as summaries in spreadsheet form, are included as Appendix D. A process schematic of the PARF is included as Figure 2.1.

[b]Elapsed time refers to the amount of time that passed before the following canister was fed into the reactor.

Table 2.4. Series 2, surrogate addition[a]

Feed event	Time	Elapsed time (min.)[b]	Notes
Can 1	11:56	0	Start surrogate feed (process data indicate Can 1 sample as retained)
Can 2	12:02	6	
Can 3	12:07	5	
Can 4	12:13	6	
Can 5	12:18	5	
Can 6	12:23	5	
Can 7	12:28	5	
Can 8	12:34	6	
Can 9	12:39	5	
Can 10	12:46	7	
Can 11	12:52	6	
Can 12	12:59	7	
Can 13	13:04	5	
Can 14	13:10	6	
Can 15	13:15	5	
Can 16	13:23	8	
Can 17	13:28	5	
Can 18	13:34	6	Severe plunger interference
Can 19	13:39	5	Feed stopped (canister did not feed)

[a]The official record of material feed and process data for all Series 1 and Series 2 test segments, as well as summaries in spreadsheet form, are included as Appendix D. A process schematic of the PARF is included as Figure 2.1.

[b]Elapsed time refers to the amount of time that passed before the following canister was fed into the reactor.

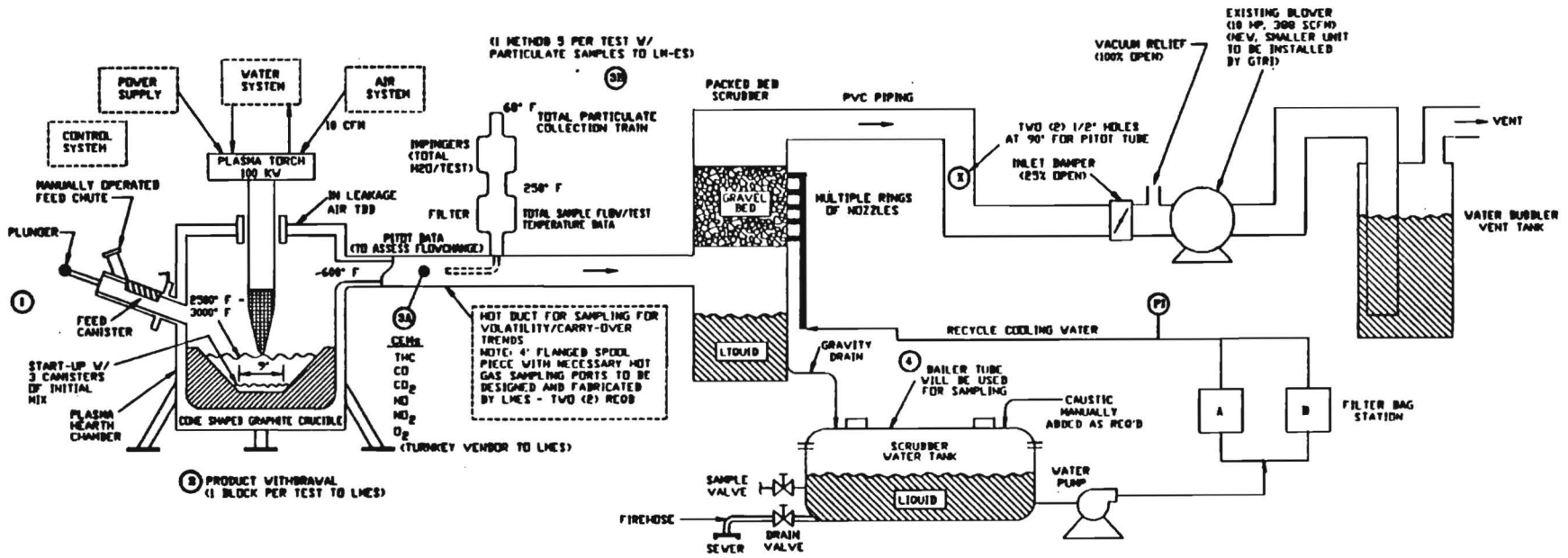


Figure 2.1. Surrogate test plan discussion figure showing sampling points.

Process parameters data manually logged during testing included:

- torch status (on/off),
- infrared pyrometer temperature (°C),
- torch position (inches, relative to internal top plane of graphite crucible),
- torch voltage (V),
- torch amps (A),
- torch carrier gas pressure (psig),
- center thermocouple—Type C imbedded in graphite crucible (°C),
- furnace pressure (inches w.c.),
- scrubber pH,
- scrubber recycle pump head pressure (psig), and
- run time (minutes).

A video of the bath was recorded for each test segment. Observations of changes with cause and effect are described in Appendix D (data sheets).

2.2 SERIES 1 AND SERIES 2 TEST DATA—PROCESS DISCUSSION

The Preheat test segment was completed to ensure all process parameters were controllable within expected ranges and to baseline the terminal temperature achievable for fixed torch operating conditions. Series 1 testing allowed for selecting the best target process conditions and operations (i.e., heat transfer, temperature benchmarks) in Series 2 testing.

Four Type C thermocouples (Figure 2.2) were imbedded in the graphite crucible. These thermocouples did not function properly in either test due to a software problem in converting the millivolt signals to temperature. Temperature data from the infrared optical pyrometer were used to establish terminal bath temperatures in the range of 1400–1600°C, at which point surrogate feed could be initiated. The pyrometer readings were initially taken by initiating torch shutdowns in an effort to reduce interference in reading accuracy as induced by the plasma itself. Thus, pyrometer readings were initially taken by applying the torch shutdown procedure and then quickly taking a reading. Based on an acute reduction in melt temperature with the torch "off" and the need to maintain a steady-state process, routine pyrometer readings were taken with the torch on-line. A separate temperature readout was used for the center Type C thermocouple for Series 2 testing. Figure 2.3 charts temperature readings for the pyrometer and center thermocouple for the duration of the run. Pyrometer readings increased from 1400°C to 1700°C while thermocouple readings rose from 1000°C to 1160°C. The pyrometer reading very quickly approached the center thermocouple reading following torch shutdown.

For each series the vessel vacuum was steady at -0.5 inch w.c., and the energy input was approximately 80 kW. The torch height varied significantly during Series 1 but was maintained at 9–11 inches for Series 2. The torch height was defined as the distance from the tip of the torch to the flat surface in the base of the crucible. (See Figure 2.24, which also indicates thermocouple locations.)

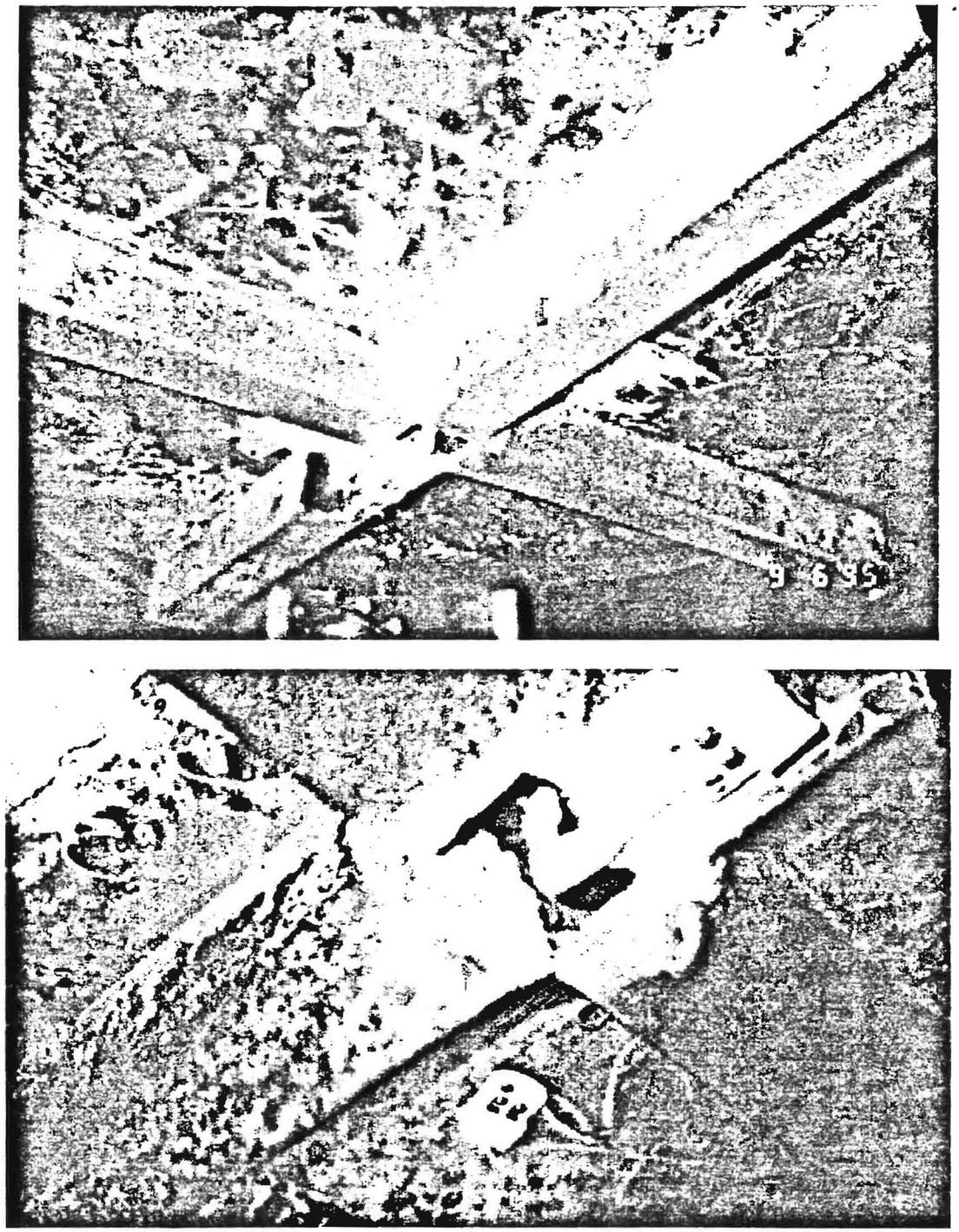


Figure 2.2. Two views of the Type C thermocouples (located beneath the crucible on the bottom reactor plate with leads embedded in the graphite, 1 inch from the melt).

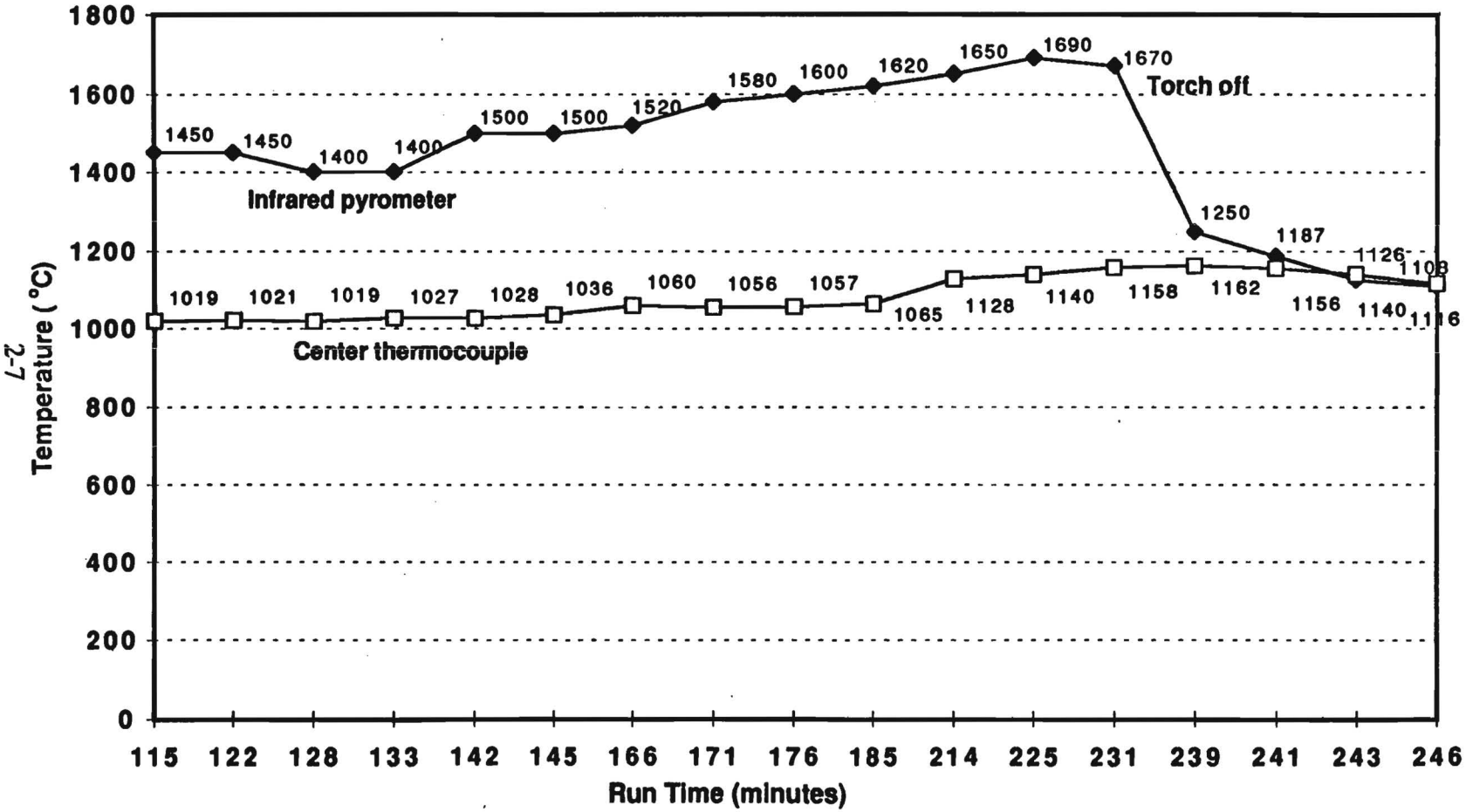


Figure 2.3. Surrogate run temperature data for Series 2.

The torch to carrier gas pressure and flow (used to vary the connection point of the arc on the electrode) varied from 35 to 55 psig (at < 10 scfm) for Series 1 and was then externally recalibrated in air (see Figure 1.1), changing the range to an optimum 25–40 psig for Series 2. The amount of iron seed melt for Series 2, introduced to the crucible during or prior to the Preheat test phase, was increased significantly over Series 1 testing to allow for a thermal mass that would quickly dissolve the feed canister contents. The relationship of iron feed quantity with melt depth for the crucible used is shown in Figure 2.5. The Series 2 molten iron depth targeted prior to Series 2 surrogate feed was 2 inches (approximately 40 lbs).

Relative to Series 1 operation, the heat-up rate and overall heat transfer/temperature profile was improved for Series 2 due to (1) increased iron bath seed material; (2) improved vessel insulation; (3) a lower torch air pressure (calibrated externally prior to Series 2) to create a flame throw approximately equal to the distance above the melt; and (4) maintaining a relatively fixed torch vertical position.

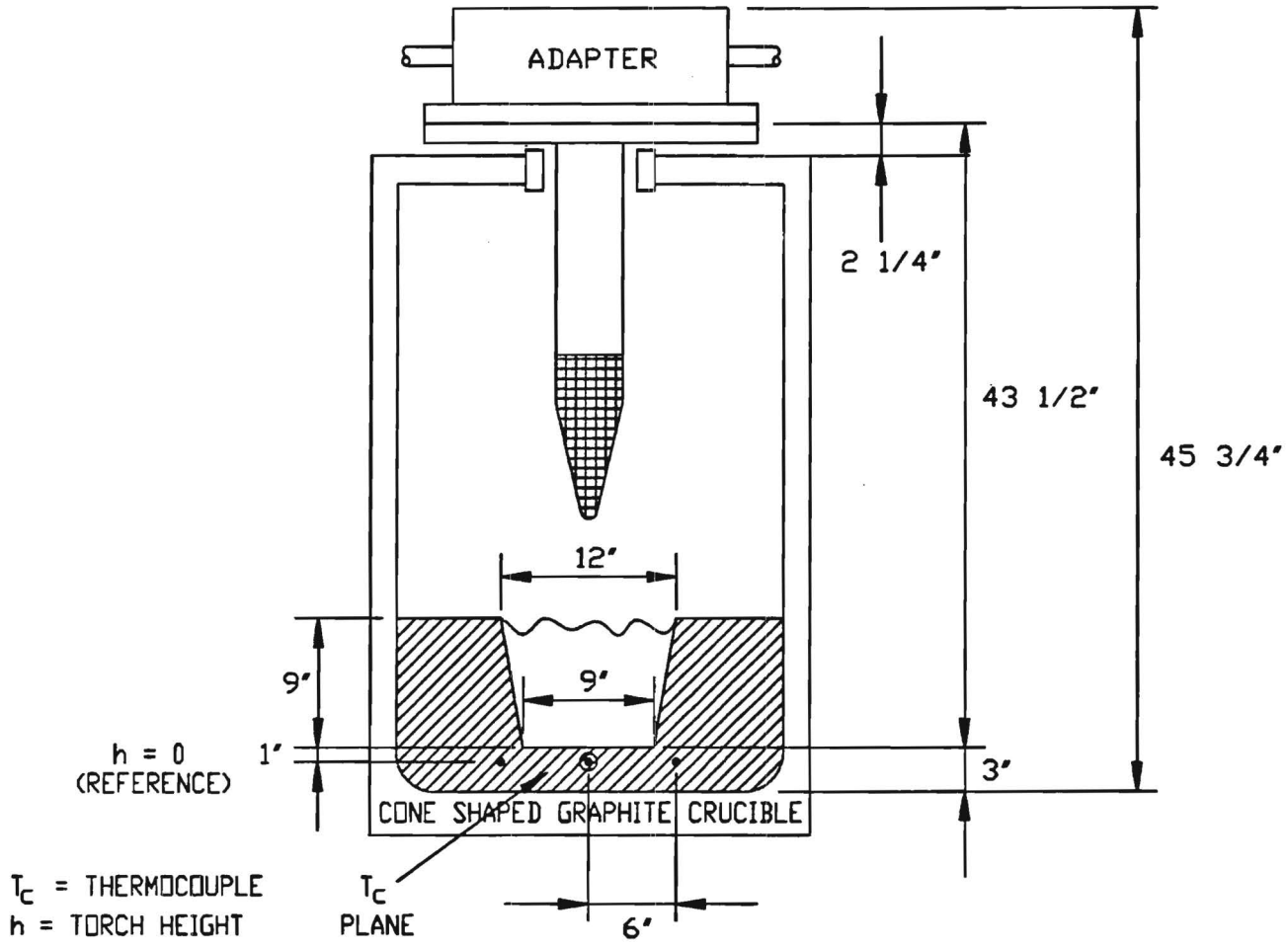
2.3 SERIES 1 AND SERIES 2 TEST DATA—CONTINUOUS EMISSION MONITORING (CEM) SUMMARY

Exhaust gas sampling from the 4-inch diameter horizontal duct exiting the reactor was performed with continuous instrumental gas analysis for oxygen, carbon dioxide, total hydrocarbons, nitrogen oxides, and carbon monoxide. Total particulate matter and volumetric flow were also measured.

Table 2.5 summarizes the continuous instrumental gas analyzers used. Sample gas to the O_2, CO_2, NO_x, and CO analyzers was conditioned to remove moisture. To avoid loss of compounds of interest due to condensation, sample gas to the THC analyzer was not conditioned. Dilution sampling at a 30:1 volumetric ratio was performed to extend the effective operating ranges of the NO_x and high concentration CO analyzers.

Table 2.5. Continuous gas analyzer instrumentation

Parameter	Detection Principle	Reference Method	Range
Oxygen	Paramagnetic	EPA 3A	0–25%
Carbon dioxide	Nondispersive infrared (NDIR)	EPA 3A	0–25%
Total hydrocabons	Flame ionization detector (FID)	EPA 25A	0–100 ppm
Nitrogen oxides	Chemiluminescent photometric	EPA 7E	0–30,000 ppm
Carbon monoxide (2 analyzers)	NDIR	EPA 10	0–1,000 ppm 0–150%



SURROGATE TEST TORCH HEIGHT

REFERENCE TO CRUCIBLE BOTTOM/ADAPTER FLANGE TOP

Figure 2.4. Schematic of the surrogate test torch with dimensions.

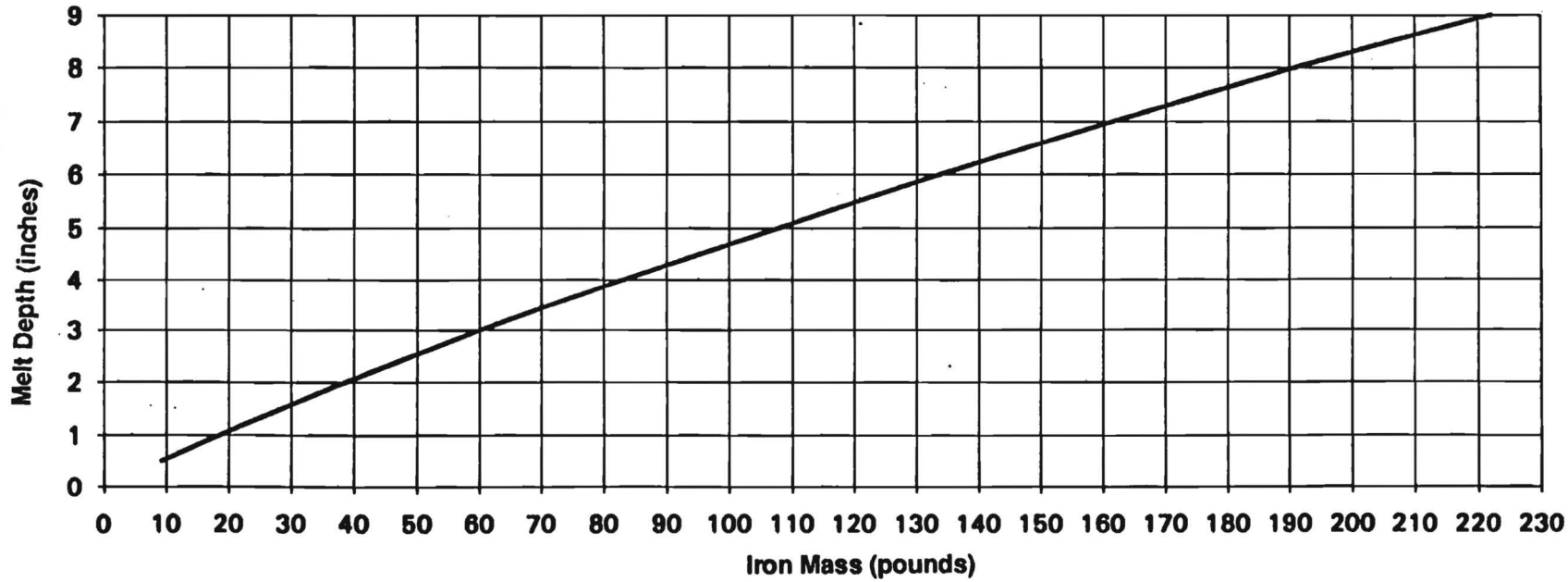


Figure 2.5. Depth of melt depicted relative to quantity of iron fed into the reactor.

A sample of particulates suspended in the exhaust gas was withdrawn isokinetically at a fixed location from the duct centerline with a stainless steel nozzle, probe, heated Teflon filter, condenser with impingers, and metering system meeting general requirements of EPA Method 5. Sample gas moisture content was calculated from impinger weight gain. Gas velocity pressure was measured downstream of the particulate sampling point with a Type-S pitot tube and inclined manometer having 0.005-inch H_2O divisions. All readings were judged to be approximately 0.005 inch H_2O. Average gas velocity for the entire duct cross section was assumed to be 0.9 times the centerline velocity.

Figures 2.6–2.13 summarize the continuous instrumental gas analyzer measurements for O_2, CO_2, THC, NO_x, and CO. Instrument readings were corrected for the initial and final system calibration check responses (for zero and upscale calibration gases).

Series 1 may be characterized by a steady consumption of available O_2 corresponding to increasing levels of CO_2 due to consumption of graphite from the crucible. Spikes of CO are notable toward the end of the run upon introduction of powdered carbon in the system feed. The NO_x levels are initially in excess of 3% and decline with decreased oxygen availability. The THC levels decline toward background after exhibiting initial values around 40 ppm.

Series 2 displays a more abrupt depletion of O_2 and levels of NO_x not quite sustained at the levels of Series 1. Spikes of CO are notable at peaks higher than during Series 1. The CO_2 trend is somewhat similar to Series 1 but with carbon consumed from system feed. The THC declines from initial levels around 20 ppm.

Table 2.6 summarizes exhaust gas characterization related to the particulate sampling and volumetric flow measurement. Standard conditions are 20°C and 29.92 inches mercury.

Included in Appendix E is a summary of the continuous instrument gas analyzer calibration responses. All documented calibrations were performed by introducing known concentration gas at the outlet of the sampling probe. Specifications comparable for environmental compliance monitoring of final effluents by the EPA reference methods would be no more than 2% calibration error for O_2, CO_2, and NO_x, a 5% calibration error for THC, and a 5% accuracy for CO measurement. Larger calibration deviations, particularly for final calibrations, may perhaps be attributed to the severe environmental conditions of the reactor exhaust gas but should not compromise the validity of evident data trends.

2.4 SERIES 1 AND SERIES 2 TEST DATA—MATERIAL BALANCE AND PRODUCT CHEMISTRY

A few brief comments regarding the Series 1 and 2 tests are appropriate in order to identify some common problems and their impacts on future systems designs. The particle size of the surrogate canister fill was 0.3 μm based on projecting a worst case scenario for thorium oxide powders formed from denitrification of thorium nitrate. In both series, difficulties were encountered in submerging the canisters into the molten bath, which resulted in splattering of the canister contents throughout the system. The photos of the final products for both test series (Figure 2.14) give an indication of the difficulties encountered with the melt. Furthermore, the data show the overwhelming predominance of iron in the vitrified melt.

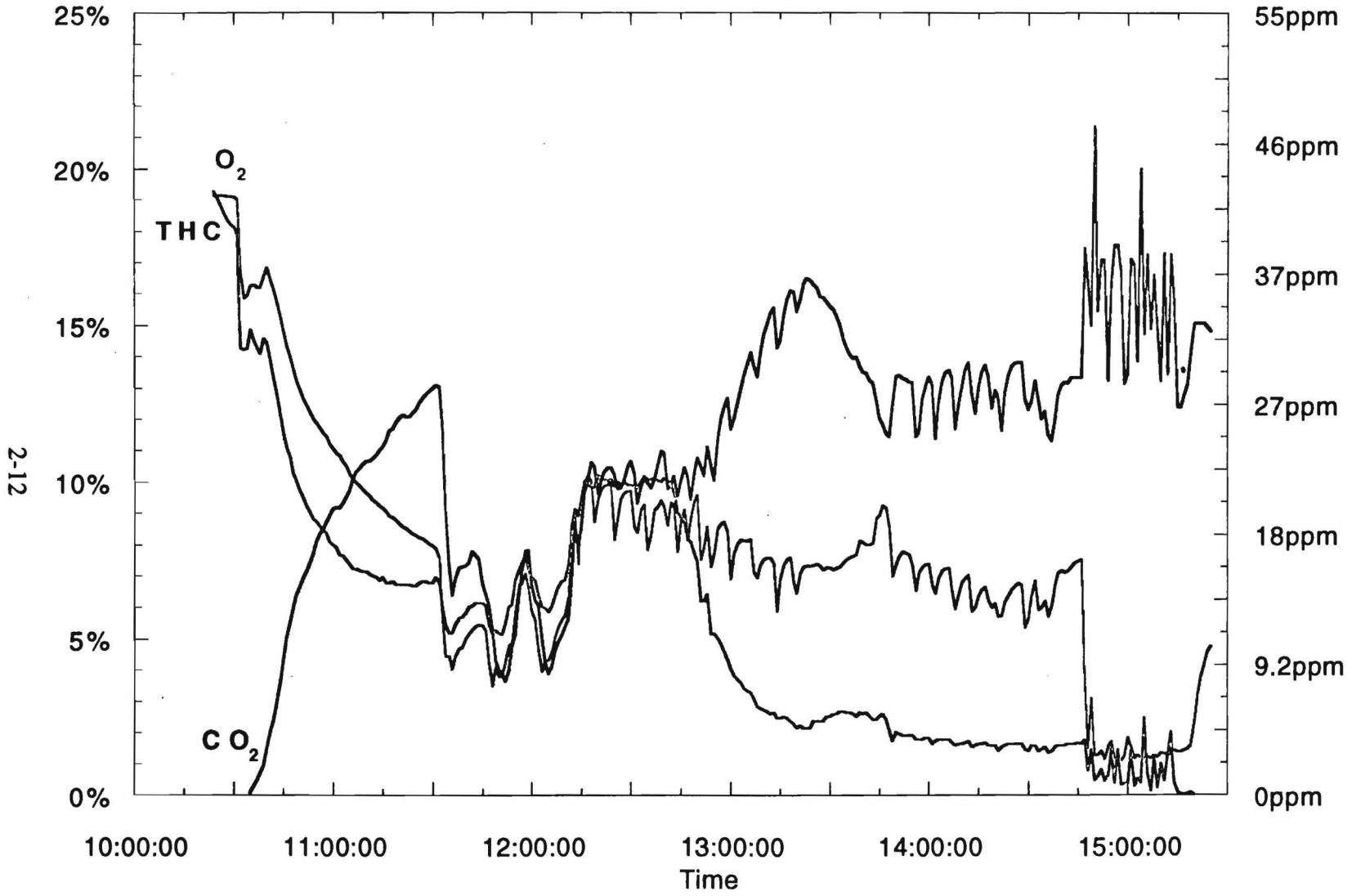


Figure 2.6. O_2, CO_2, and THC data from Series 1.

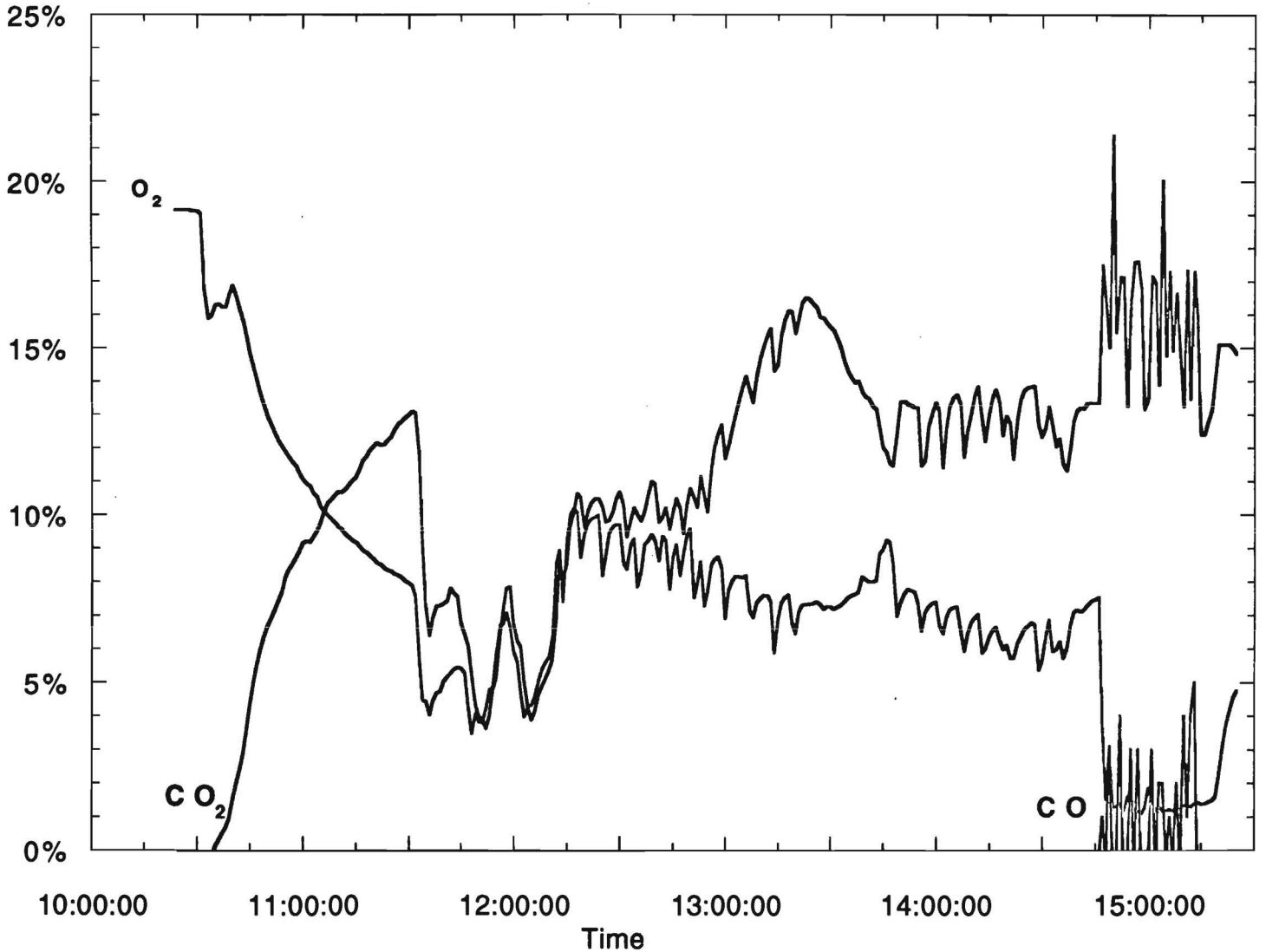


Figure 2.7. O_2, CO_2, and CO data from Series 1.

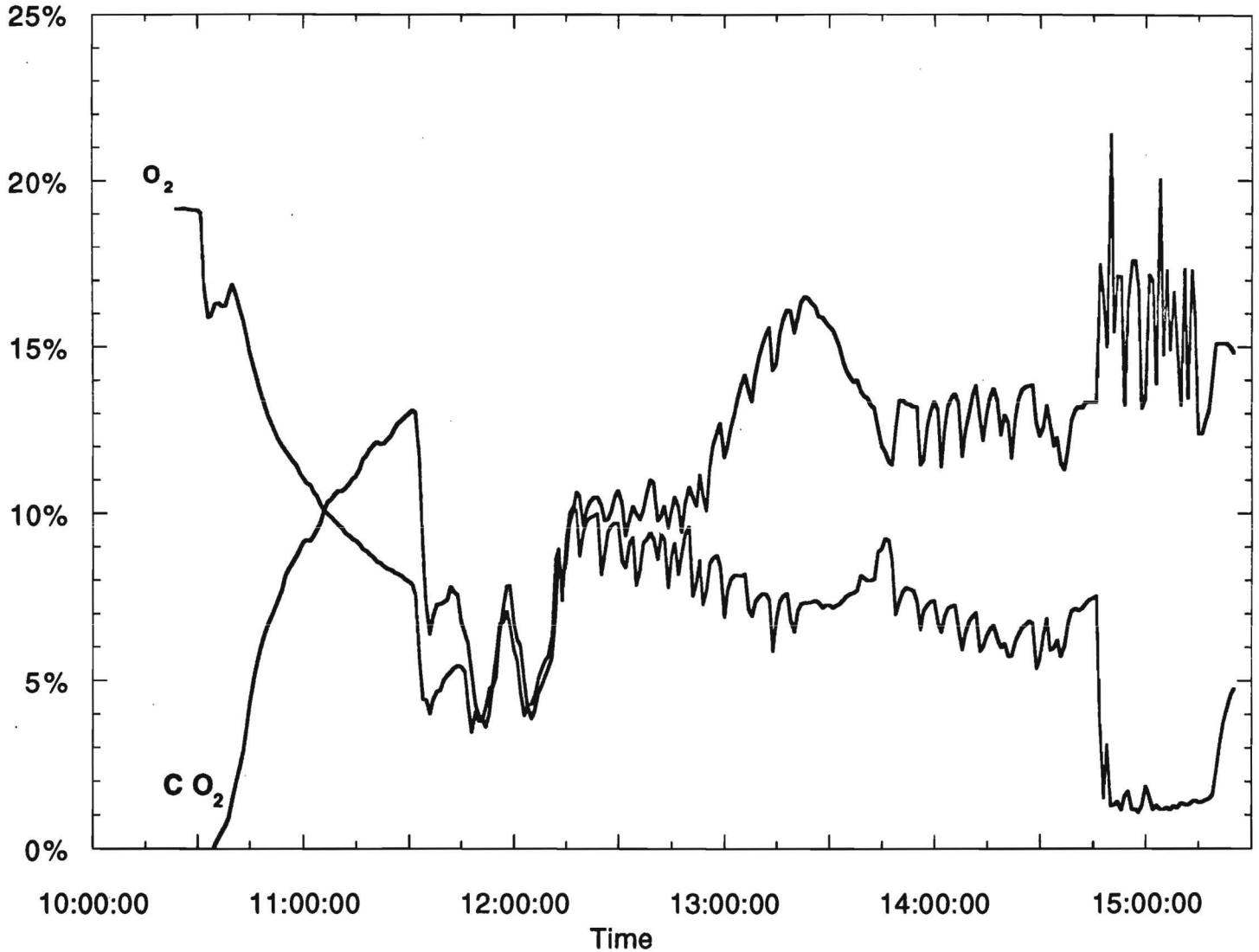


Figure 2.8. O_2 and CO_2 data from Series 1.

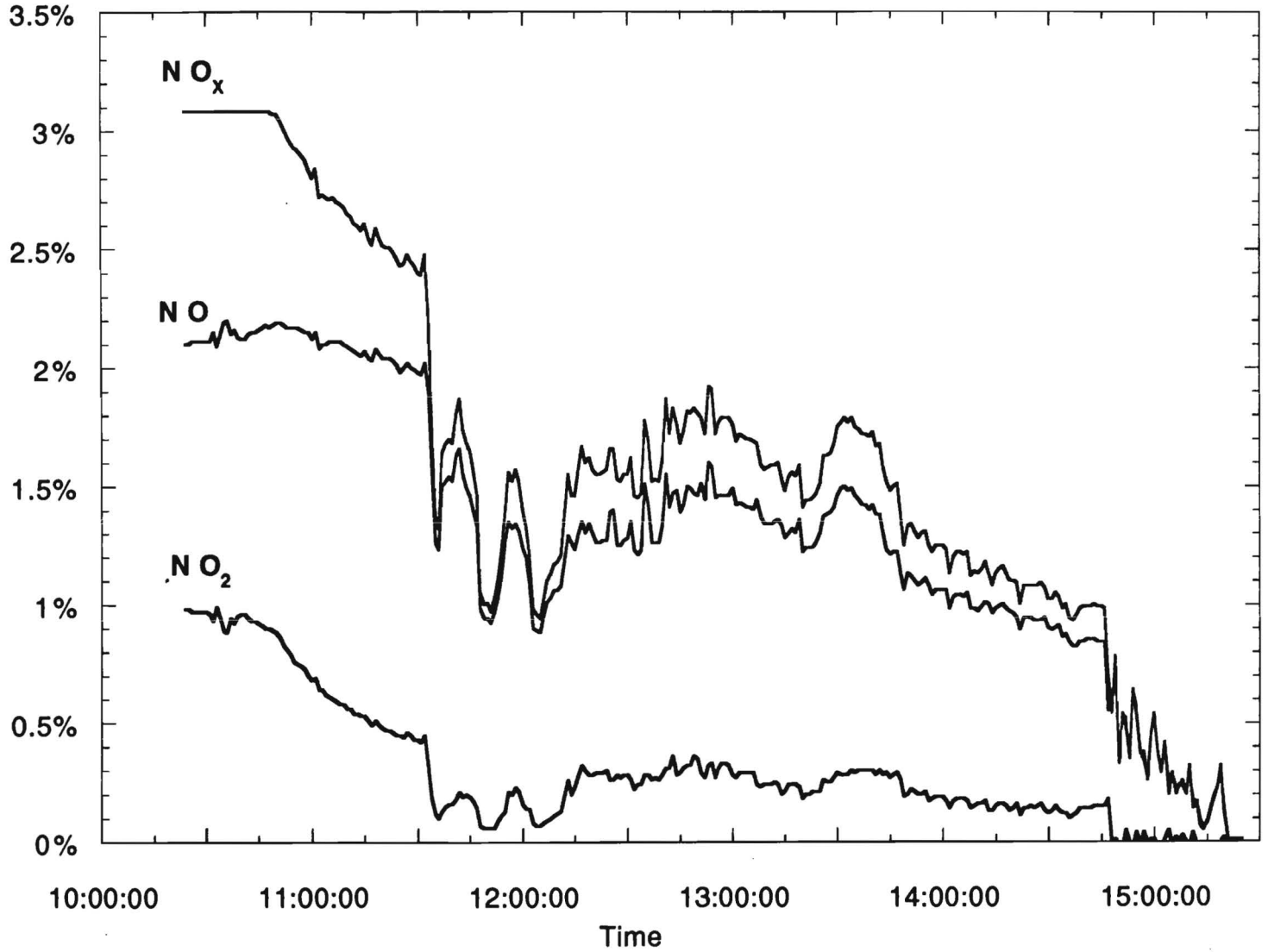


Figure 2.9. NO_x, NO, and NO_2 data from Series 1.

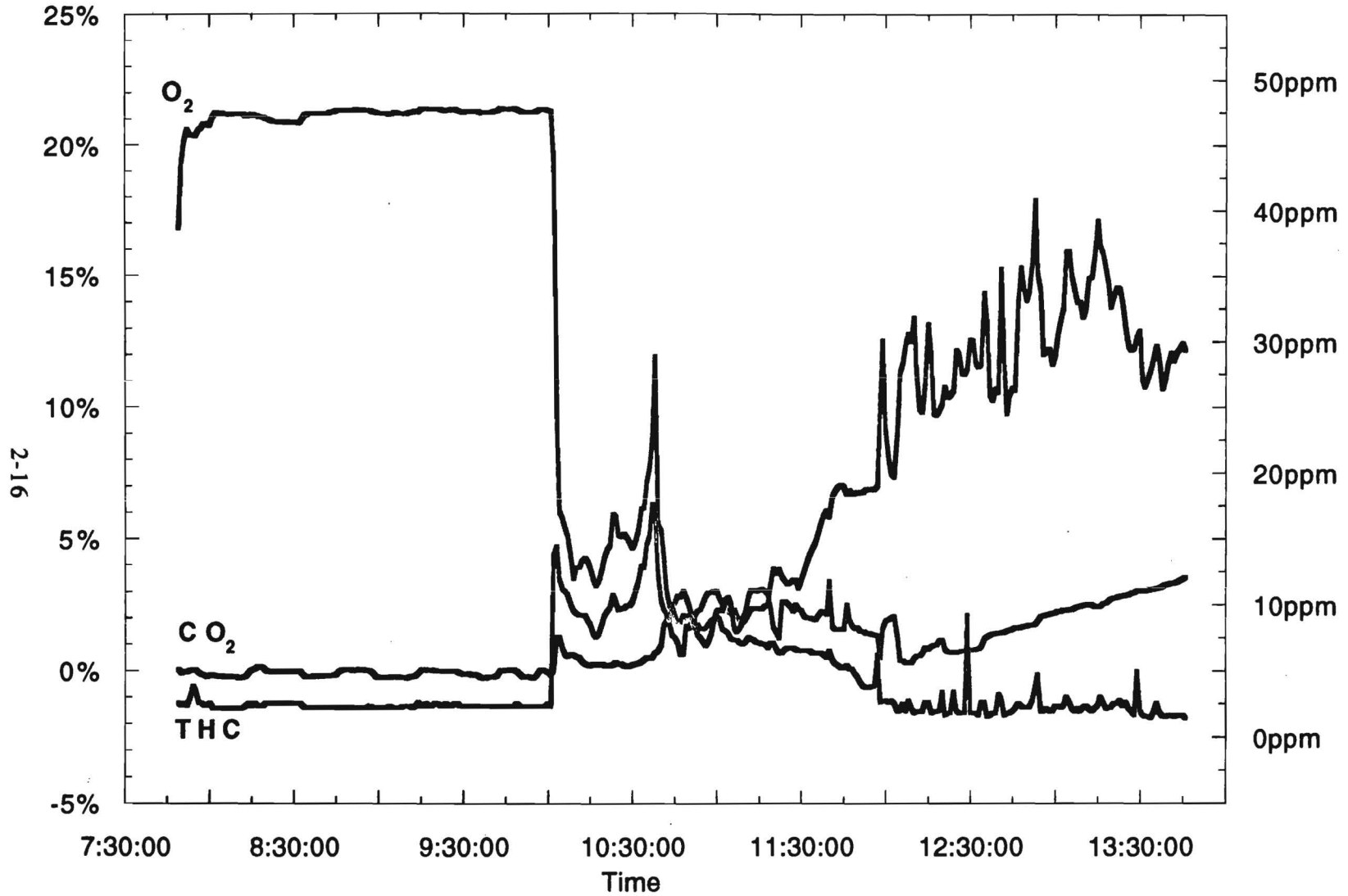


Figure 2.10. O_2, CO_2, and THC data from Series 2.

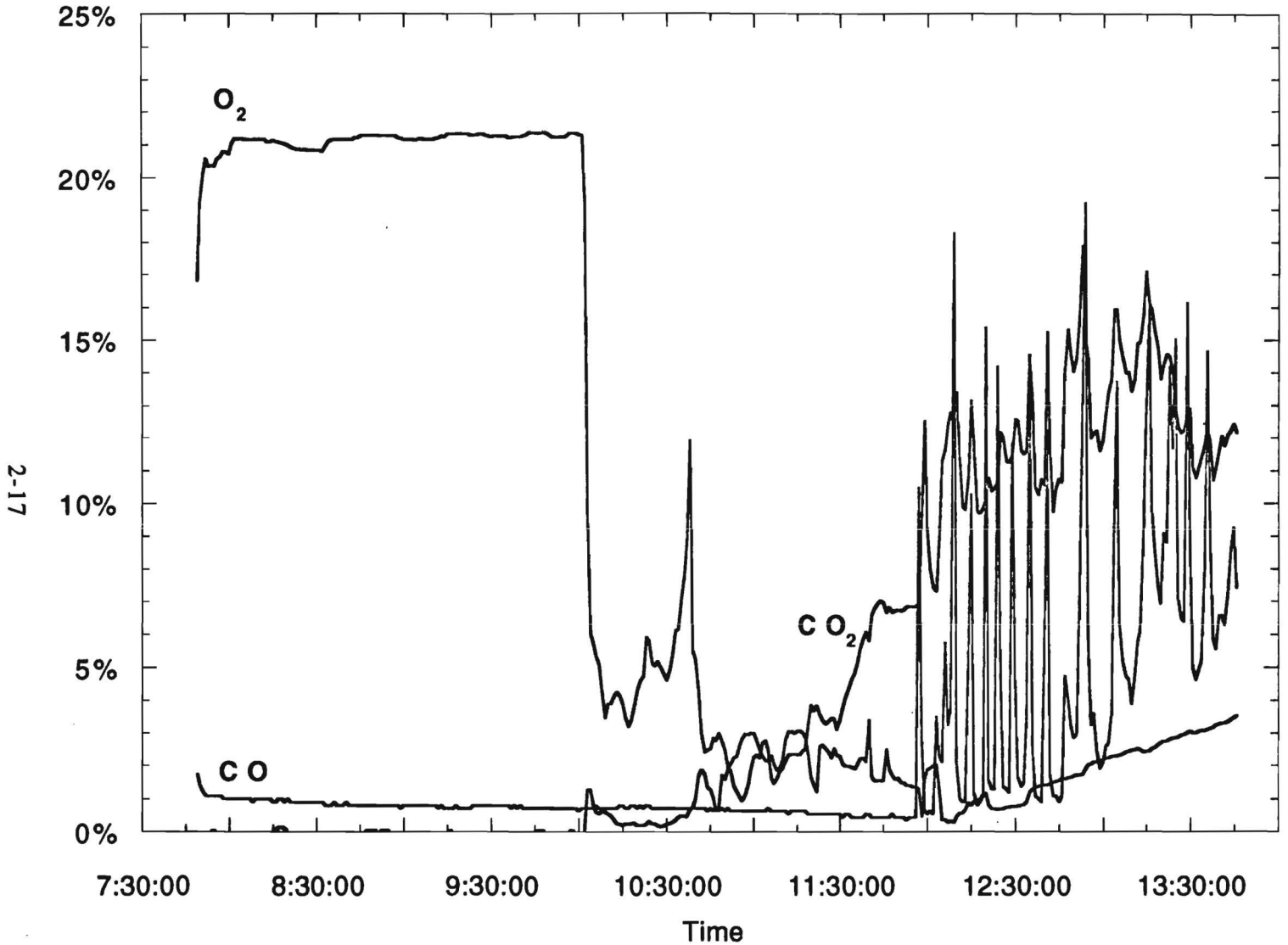


Figure 2.11. O_2, CO_2, and CO data from Series 2.

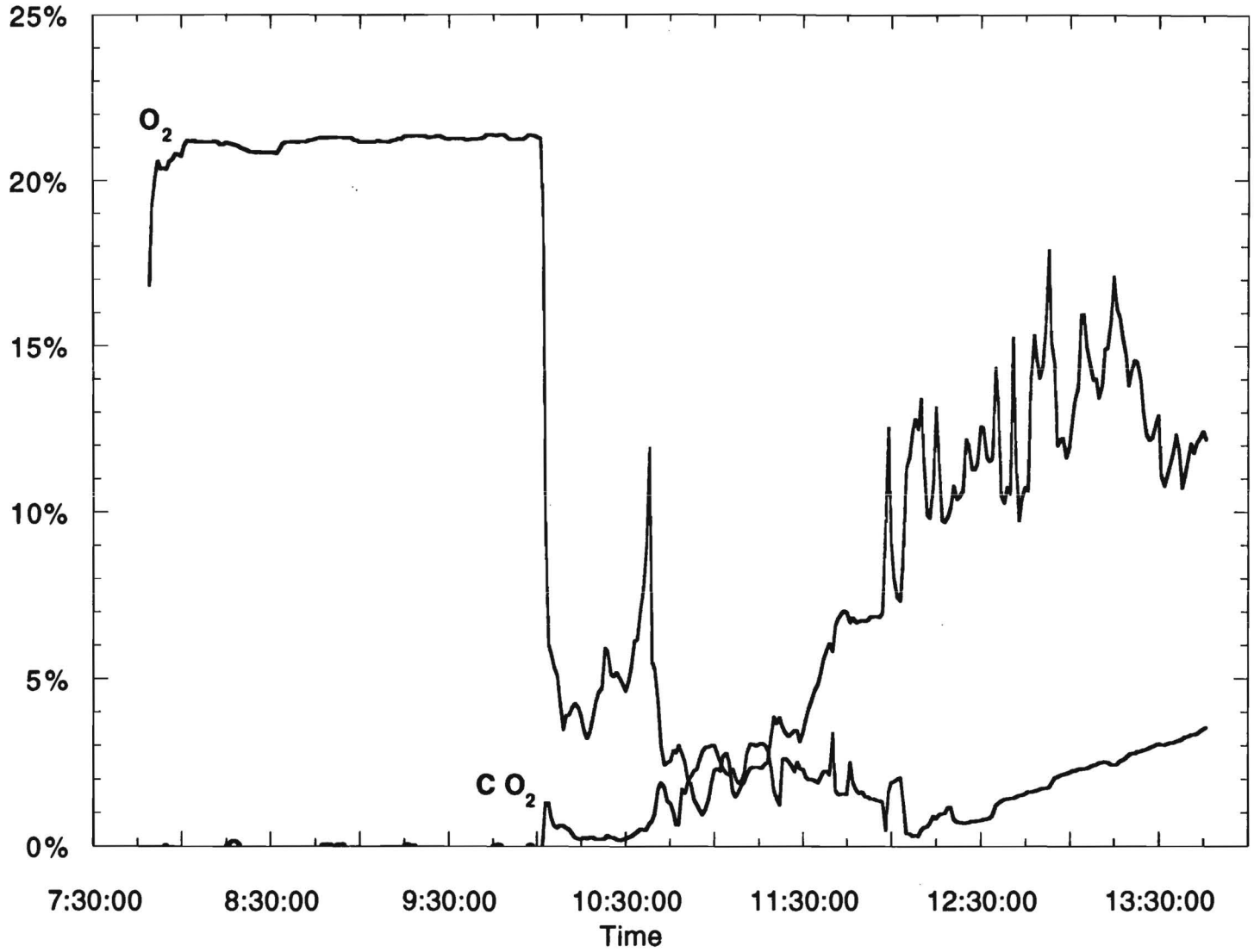


Figure 2.12. O_2 and CO_2 data from Series 2.

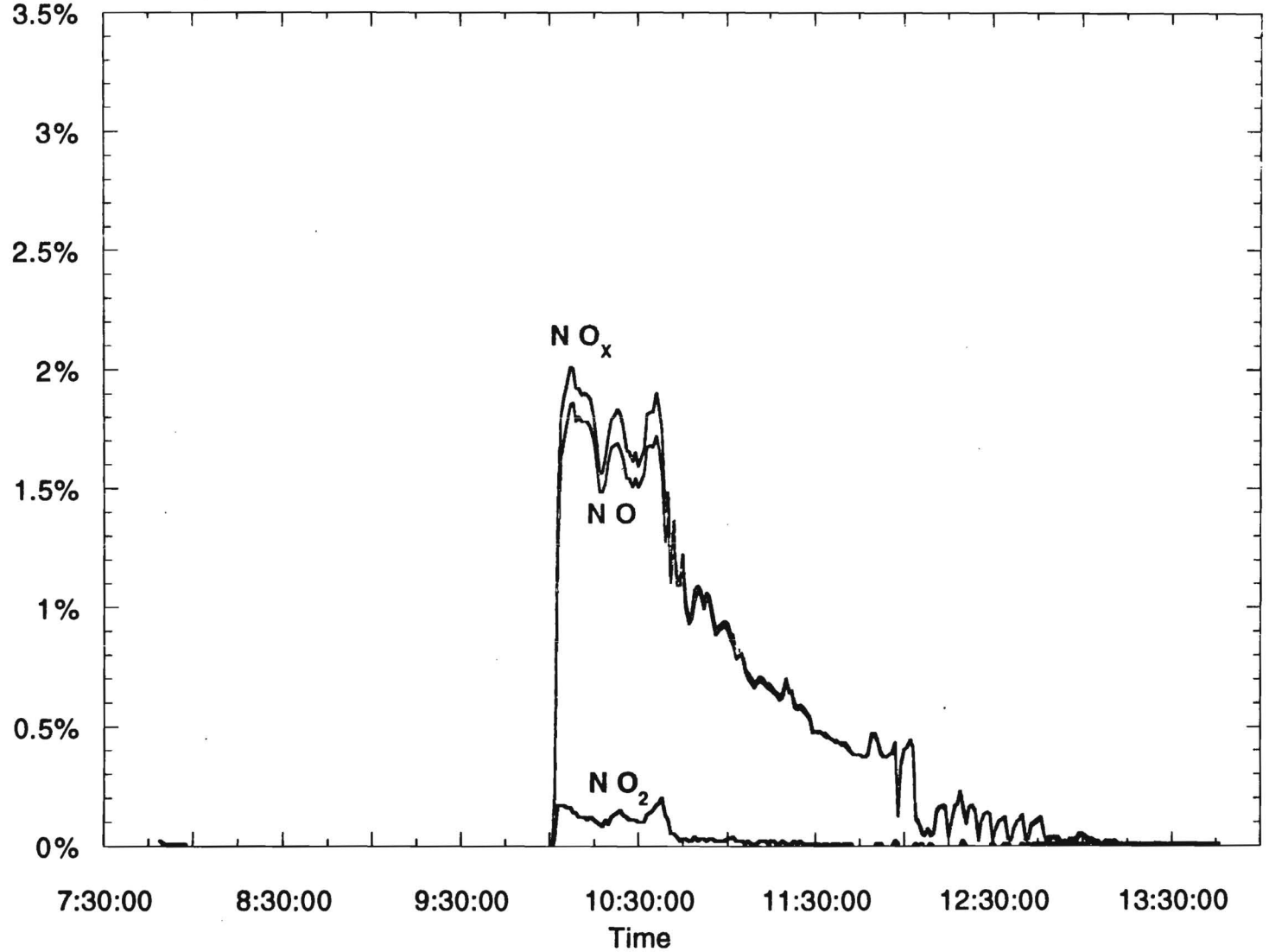


Figure 2.13. NO_x, NO, and NO_2 data from Series 2.

Table 2.6. Particulate sampling and flow measurement summary

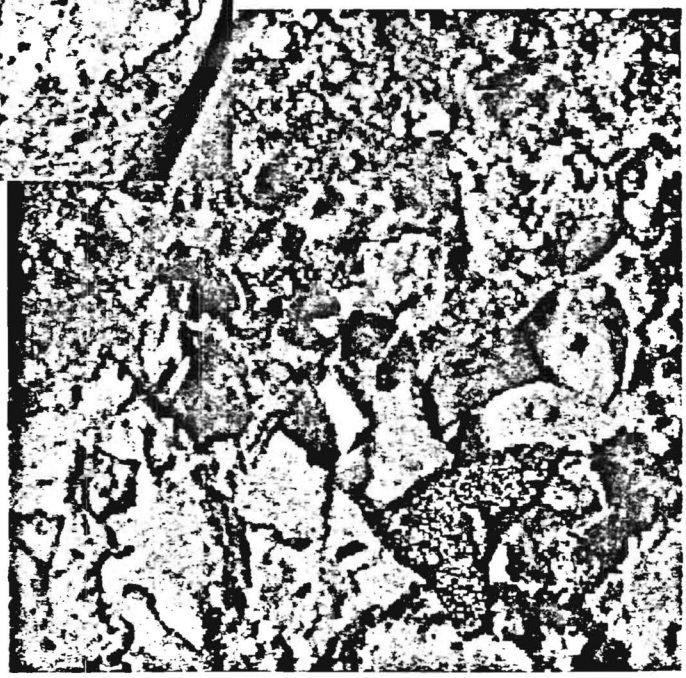
Parameter	Units	Series 1	Series 2
Date	calendar	09/12/95	09/19/95
Start time	clock	12:12	11:59
Stop time	clock	15:17	13:29
Static pressure	inches H_2O	-0.4	-0.4
CO_2	dry volume %	13.1	12.5
O_2	dry volume %	6.6	1.6
NO	dry volume %	1.1	0.075
NO_x	dry volume %	1.3	0.08
THC	ppmv	7.9	2.3
CO	dry volume %	peaks near end of run	5.4
Moisture	volume %	2.1	1.4
Duct temperature	°C	239	171
Gas velocity	actual ft/s	5.2	4.9
Volumetric flow	dscfh[a]	798	866
Gas sample volume	dscf[b]	101.4	48.8
Percent of isokinetic sampling	percent	97	97
Particulate catch (probe + filter)	grams	15	7
Nozzle Diameter	inches	0.7525	0.7482

[a]dry standard cubic feet per hour
[b]dry standard cubut feet

A major cause for the difficulty in submerging the canisters is the high density and viscosity of the molten iron bath. These observations reveal a need to modify the bath chemistry so as to 1) lower the density and perhaps the melt temperature and 2) lower the viscosity. Such bath changes should aid in complete submergence of the canisters and reduce the amount of particulate matter carried downstream. A second method for achieving more desirable bath characteristics that was considered as an outgrowth of these test results would be to eliminate iron completely by removing the drum contents and processing them in a nonmetallic bath.

Series 1

Series 2



Series 2 closeup

Figure 2.14. Final products from Series 1 (top photo) and Series 2 tests.

Finally, Series 2 may offer important data pertaining to what may occur in the testing of thorium oxide (ThO_2) with CaO under similar test circumstances since both $CaO–ZrO_2$ and $CaO–ThO_2$ binary systems contain 1:1 compounds such as $CaZrO_3$ and $CaThO_3$, respectively. As was evident in the Series 2 test, $CaZrO_3$ formed readily, utilizing all available ZrO_2; therefore, one might expect similar results for the $CaO—ThO_2$ system. That is, $CaThO_3$ will form, and there should be an excess of CaO if the quantities of CaO and ThO_2 are in agreement with the simulated drums analyses. These observations suggest a final product of $CaThO_3$ rather than ThO_2 after vitrification.

2.4.1 Material Balance, General Chemistry, and Particle Size

Figure 2.15 is a schematic diagram of the plasma vitrification test apparatus. Points A, B, and C identify the locations within the system from which test products were obtained and subsequently analyzed. The following discussion pertains to issues related to these locations. Point A is the location for the final vitrified product within the graphite crucible. Analysis of that product will determine the extent and nature of the surrogate interaction with the other "drum" constituents. The material at point B is not part of the vitrified product but has not escaped the primary chamber and will represent a build-up and removal hazard for a production process. Understanding what this material is and why it was not incorporated into the final product will help to optimize the high temperature process. Finally, the off-gas solids located at C represent the material that must be prevented from escaping the system through the air pollution control (APC) system. Understanding the nature of this material will help to ensure that the APC design will fully protect the workers and the environment.

2.4.1.1 Series 1

Figure 2.16 is a schematic diagram of the plasma vitrification system and serves to identify the locations of the products. Samples collected from point 1 were split into three sub-samples: 1) the solid fused product, 2) loose material <0.85 mm in diameter, and 3) loose material >0.85mm in diameter. Samples collected at points 2 and 3 were also subdivided into fractions less than and greater than 0.85 mm in diameter. In Table 2.7 are listed the samples, their collection points, and their respective weights (in grams). The total weight of all components (19,944 g) was compared to the weight of the feed material (15,978 g), which corresponds to a 24.8% increase in weight for the reaction product. Much of this increase in weight can be attributed to the oxidation of the iron.

The data presented in Table 2.8 are the X-ray diffraction (XRD) analyses of those samples identified in Table 2.7, with the exception of the main body, which will be discussed in greater detail in the following section. The relative intensities of the diffraction patterns correspond to phase concentrations in the samples.

Samples from area 1 reveal significant variations between the finer and coarser particles in that (per author) the latter are rich in calcium compounds such as CaO, $CaCO_3$, and $Ca(OH)_2$, whereas these compounds are nearly absent in the former. However, in both groups MgO, Fe_3O_4, and Fe_2O_3 are strongly represented. Location 2 yielded particles rich in MgO as well as Fe_3O_4 and Fe_2O_3 much $ZrSiO_4$ associated with the >0.85 mm particles. The presence

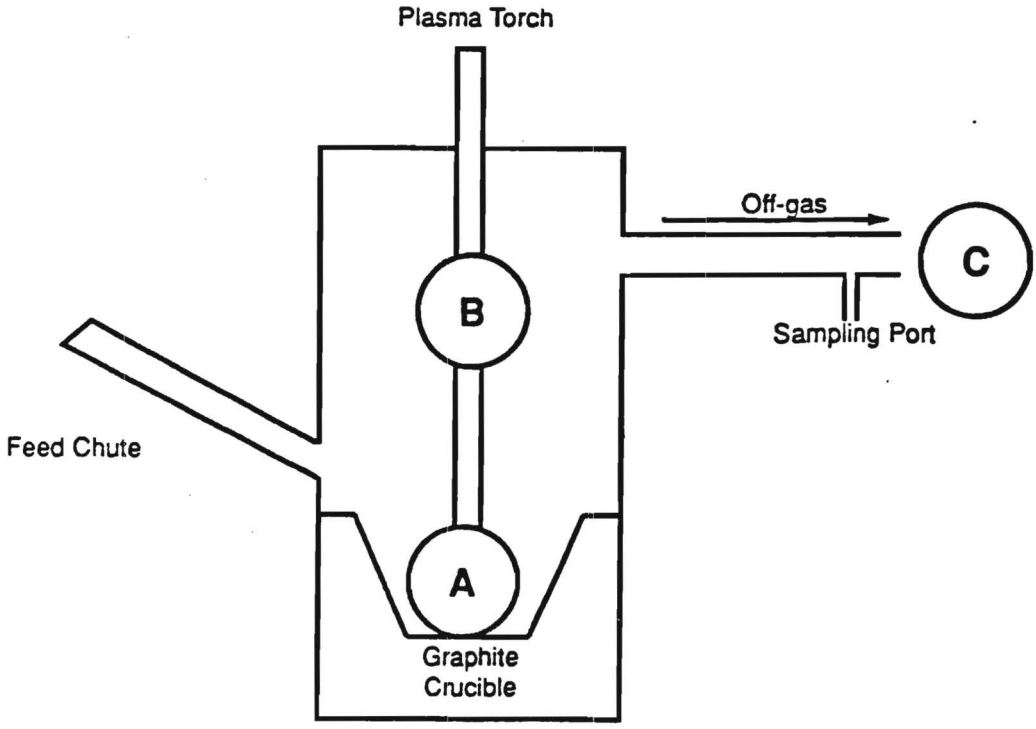


Figure 2.15. Schematic diagram of plasma vitrification system.

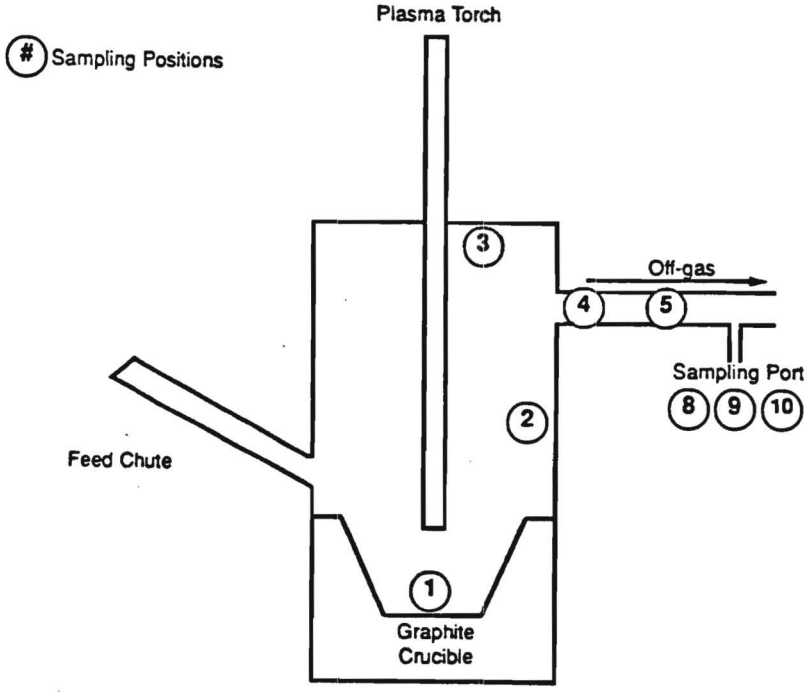


Figure 2.16. Schematic diagram of plasma vitrification system with the locations for the products from Series 1 test enumerated.

of $ZrSiO_4$ in this sample is due to its use as a mold parting agent that is painted onto the graphite crucible walls. Also associated with the graphite crucible is the graphite component found throughout these samples. In general MgO, Fe_3O_4, and Fe_2O_3 are the dominant phases identified in locations 3–10, with some lesser amounts of $CaCO_3$, $Ca(OH)_2$, and graphite.

Sample S1-8 was found to have regions rich in lead and chlorine confirmed by XRD to be PbOCl. EDS analyses of the weld region of the steel canisters revealed lead as a component and the probable source of lead on the air filter, due to its low melting point. Also worth noting is the absence of CeO_2, which had been added as a tracer to the feed material. It may be that the dissemination of CeO_2 throughout the products resulted in concentration levels too low to detect.

2.4.1.2 Series 2

The collection points from which the Series 2 test products were obtained is shown in Figure 2.17. However, unlike the products collected for the Series 1 test, these samples were not subdivided on any basis. Table 2.9 relates the weight of these products to their collection points. A comparison of total product weight (26,754 g) with the feed weight of 23,340 g shows a weight increase of 14%. As in Series 1, the weight gain is most likely due to oxidation of the iron. The difference in the percentage weight gain between the two series may be explained on the basis of the level of oxidation in Series 2, where a large mass of elemental iron exists as a layer and rim. A crude calculation as to the mass of the iron layer present suggests at least 4% of the original iron feed is present as elemental iron while all remaining iron is present as FeO, which should result in a smaller weight increase in Series 2 test products.

Table 2.7 contains the identity and concentration of the various phases corresponding to their locations in the vitrification system. The compounds FeO, Fe_3O_4, and $CaZrO_4$ are the prominent phases detected in samples from locations 1, 2, and 3. Samples from points 5–8 contain concentrations of $CaZrO_4$ and $CaCO_3$/CaO with much smaller amounts of iron oxides and graphite. In this respect there are similarities between Series 1 and 2 in that the off-gas samples are poor in iron oxides and rich in surrogate materials.

2.4.2 Characterization of Macrostructures and Microstructures in the Vitrified Product Melts

2.4.2.1 Series 1

Figure 2.18(A) is an optical micrograph of a cross section of the vitrified melt from Series 1 after removal from the graphite crucible and corresponds to location A shown in Figure 2.15. This micrograph serves to illustrate the varying macrostructures that evolved during heating and subsequent cooling of the plasma-melted mixture. The region interfacing the graphite crucible solidified in a manner analogous to chill casting resulting in elongated grains growing normal to the chill surface (i.e., the graphite crucible contact zone). This outer skin also appears to contain a greater amount of small pores probably due to the CO_2 gas bubbles that formed from the carbon (graphite) and oxygen reactions at the interface. In contrast, the macrostructure of the inner region of the micrograph consists of a more uniform, massive structure.

Table 2.7. Weight distribution (in grams) of product per collection point

Sample	Sample description	Weight (grams)	Percent of total	Subgroups
S1-1a	Main Product	16,453	82.50	17,372 g (87.1%)
S1-1a	Side walls (loose)	919	4.61	
S1-1b	Inside loose <0.85 mm	183	0.92	495 g (2.48%)
S1-1c	Inside loose >0.85 mm	312	1.56	
S1-2b	Main body deposits <0.85 mm	392	1.97	548 g (2.75%)
S1-2c	Main body deposits >0.85 mm	156	0.78	
S1-3b	Top cap deposits <0.85 mm	690	3.46	1321 g (6.62%)
S1-3c	Top cap deposits >0.85 mm	631	3.16	
S1-4b	Off-gas arm deposits	67	0.34	
S1-5b	Spool piece deposits	67	0.34	208 g (1.04%)
S1-8b	Air filter	10	0.05	
S1-9b	Off-gas powder	59	0.30	
S1-10b	Air filter probe	5	0.03	
	Totals	19,944	100.02	N/A

Table 2.8. X-ray diffraction results for Series 1 testing—primary phases observed[a]

Sample	MgO	CaO	$CaCO_3$	$Ca(OH)_2$	Fe_3O_4	Fe_2O_3	$ZrSiO_4$	Graphite
S1-1B-1	VS	W	W	W				
S1-1B-2	S	W	W		S	M		
S1-1C-1	VS				S	M		
S1-1C-2	M	VS	MS	M				
S1-1D	VS							S
S1-2B-1	VS		W					MS
S1-2B-2	VS		W		MS	M		M
S1-2C-1	MW						VS	
S1-2C-2	VS				S	VS	M	M
S1-3B-1	VS		W					W
S1-3B-2	S		W		S	S		
S1-3C-1	VS							W
S1-3C-2	M				S	S		
S1-4-1	VS		W					M
S1-4-2	M				S	MS		
S1-5	VS							W
S1-8[b]	S		M	M				W
S1-9-1	VS							S
S1-9-2	S							VS
S1-10	VS		MW					W

[a]VS—very strong; s—strong; ms—medium strong; m—medium; mw—medium weak; w—weak

[b]This sample contains a small amount of PbOCl

Table 2.9. Weight distribution of product per collection point

Sample	Sample description	Weight (g)	Percent of total	Subgroups
S2-1a	Main Product	13,930	52.07	15,172 g (56.7%)
S2-1b	Crucible side walls	1,242	4.64	
S2-2	Inside loose	1,524	5.70	1,524 g (5.7%)
S2-3	Main body deposits	9,580	35.81	9,580 g (35.8%)
S2-4	Top cap deposits	431	1.61	431 g (1.6%)
S2-5	Spool piece deposits	39	0.15	
S2-6	Air filter probe	2	0.01	47 g (0.2%)
S2-7	Air filter	5	0.02	
S2-8	CEM filter	1	0.00	
Totals		26,754	100.01	N/A

Detailed analyses of the various macrostructures within the cross section obtained by combined scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), and XRD yielded related microstructures, chemistry, and phase identities, respectively. The SEM and EDS data were accumulated for areas identified as crust and interior regions, as shown in Figure 2.18(A). In addition, X-ray elemental maps for select microstructures served as an aid in phase identification. Two microstructures and representative EDS analyses were chosen to illustrate the typical structure for the regions within the cross section designated as the crust region and the interior region.

The crust region forms the outer rim of the cross section and, as previously noted, consists of material that was in direct contact with the graphite crucible. The microstructure [Figure 2.18(B)] appears to be homogeneous with no discernible grains, only numerous pores left by gas bubbles. The EDS data [Figure 2.18(C)] reveals iron as the principal element with only traces of manganese and silicon. Consistent with the EDS data are the XRD analyses [Figure 2.18(D)], which show Fe_3O_4 and FeO as the sole phases. It would appear that solidification of the rim mixture had preceded the distribution of MgO and CaO into the melt.

Table 2.10. X-ray diffraction results for Series 2 testing—primary phases observed[a]

Sample	ZrO_2	$CaZrO_3$	CaO	$CaCO_3$	$Ca(OH)_2$	FeO	Fe_3O_4	Fe_2O_3	C
S2-1a		MW				VS			VW
S2-1b		W				VS	W		
S2-2		MW				S	S	W	
S2-3		MW				S	MS	W	
S2-4	M			MS		W	S	S	
S2-5	W	M		S		W	MW		VW
S2-6		MW		MW	M	MW	MW	W	VW
S2-7		M	MS			W	S		VW
S2-8		W		MS		M	VS		W

[a]VS—very strong; s—strong; ms—medium strong; m—medium; mw—medium weak; w—weak

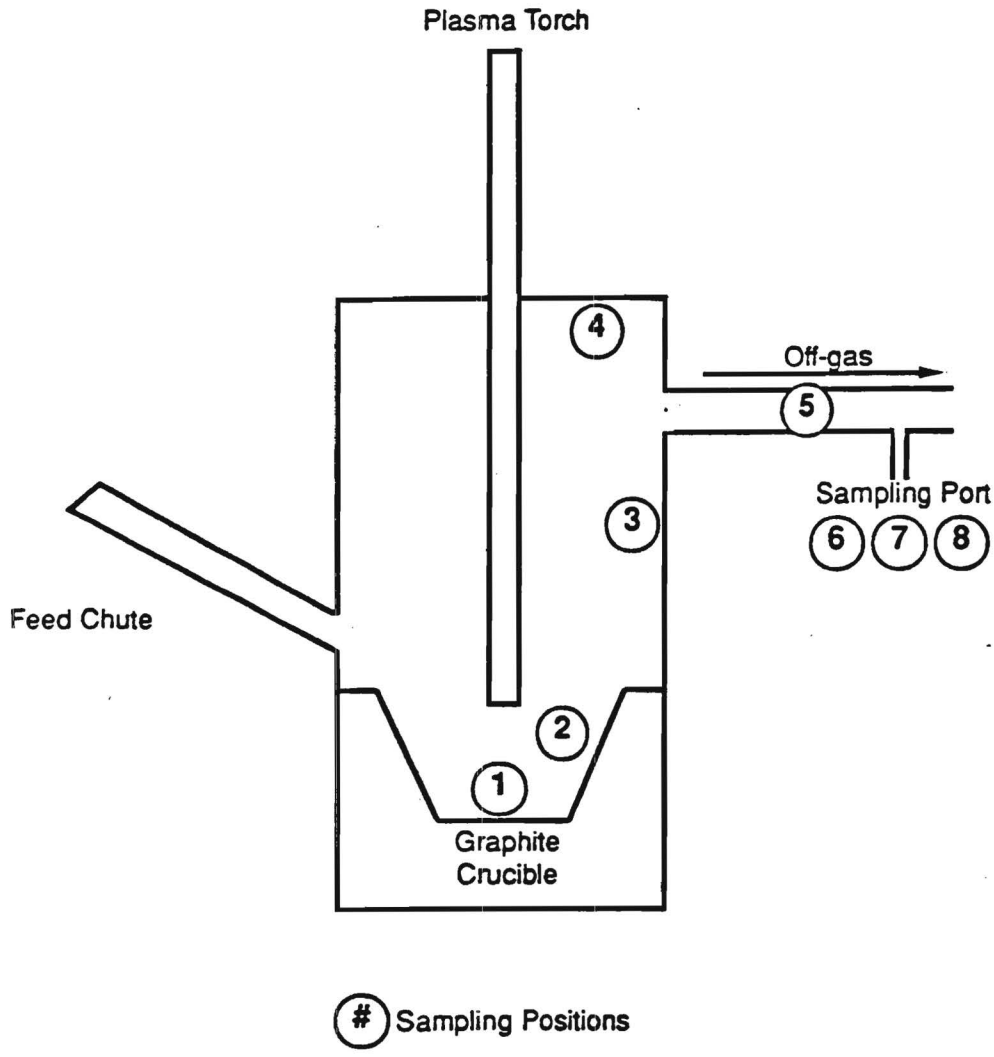


Figure 2.17. Schematic diagram of plasma vitrification system with the locations for the products from Series 2 test enumerated.

Support for this thesis comes from our observation of entrapped MgO powders that form a veinlet (Figure 2.19) between the outer rim and the material immediately above, which was sufficiently cooled so that no reaction had occurred between the MgO and molten iron oxide. A vienlet of unreacted material indicates a bath temperature difference existed. It is important to note that XRD analyses failed to reveal free iron, which indicates the oxidation of iron occurred throughout the crucible material.

Figure 2.18(E) is a microstructure of an area within the interior region. The dominant feature in this microstructure is the irregularly shaped dark gray grains surrounded by a matrix of light gray phases. Bulk EDS of this microstructure [Figure 2.18(F)] reveals iron, calcium, magnesium, and oxygen as the primary elements, with iron being predominant. XRD data [Figure 2.18(G)] obtained from two separate samples from the interior region served to identify four crystalline phases, namely, $CaFe_3O_5$, $Ca_2Fe_2O_5$, Fe_3O_4 and (Mg,Fe)O.

At higher magnifications [e.g., Figure 2.20(A)], the nature of the dark gray grains and their immediate surroundings can readily be seen. X-ray dot maps for the four major elements were obtained to aid in phase identification. These maps, in combination with XRD data, permit identification of phases within the microstructure. Analysis of components within the grains reveals the light dendritic phase as dominantly an iron oxide [Figure 2.20(B)] while the darker surrounding phase is rich in magnesium [Figure 2.20(C)]. It would appear that the dendritic phase is iron oxide, most probably Fe_3O_4, and that the darker phase may be (Mg,Fe)O and/or $MgFe_2O_4$. These latter choices are based on the absence of MgO in the XRD patterns.

2.4.2.2 Series 2

Prior to discussing the results of the tests for Series 2, a brief restatement of test differences between Series 1 and 2 is worth noting (e.g., 18.7 kg of iron were placed into the graphite crucible in the pretest for Series 2, as compared to only 7 kg in Series 1). This was done in order to increase the volume of molten metal into which the canisters with the surrogate mixtures would be introduced for melting, based in part on observations made in Series 1 testing. In addition, the position of the torch for Series 2 was more restricted (i.e., closer to a fixed position), and finally, the compositional differences (see Table 1.2) in these series are apt to play a role in such tests.

Figure 2.21(A) is an optical micrograph of a cross-section segment from the solidified melt of Series 2. This macrostructure serves a function similar to that of Figure 2.18(A) in Series 1, namely, to illustrate the various structures that evolved during heating (melting) and the subsequent cooling of the Series 2 mixture. Several features in this cross section differ significantly from those observed in Series 1 [Figure 2.18(A)]. For example, a metallic rim formed at the interface between the molten iron and the graphite crucible wall, and a metallic-looking layer, approximately 12 mm thick, is present at the bottom of the solidified melt. The nature of those metallic regions was determined by means of chemical analyses (EDS) and XRD, as illustrated in Figure 2.21 (B and C, respectively). In all instances, the metallic materials consist primarily of iron with trace amounts of manganese and silicon. The XRD data were found to be in excellent agreement with data for iron.

Subsequent characterization of the bulk interior regions by EDS and XRD is shown in Figure 2.21 (D and E, respectively). The elemental chemistry of this region consists of iron, calcium, and zirconium. The primary phases, as obtained from several areas throughout the interior region, consisted primarily of FeO and $CaZrO_3$, with some areas showing trace amounts of ZrO_2 as well as unidentified lines. It is important to note that the quantities of CaO and ZrO_2 composing the mixtures in the canisters should have yielded a surplus of CaO over that required to form the zirconate ($CaZrO_3$). However, no free CaO or calcium ferrates were observed, nor were ferric oxides such as Fe_2O_3 or Fe_3O_4, suggesting a more reducing environment for Series 2 than for Series 1. It is possible that some CaO was incorporated in FeO since it is known that FeO can include up to 30 mole percent CaO at temperatures of 1100°C, and that if rapid cooling occurs, the CaO will be retained in the FeO structure. Possible indications of such an occurrence were suggested by the presence of an extremely weak Ca peak in the EDS analyses of the FeO phase. If CaO is contained within the FeO phase even at the 1-wt % level, some of the excess CaO could be accounted for.

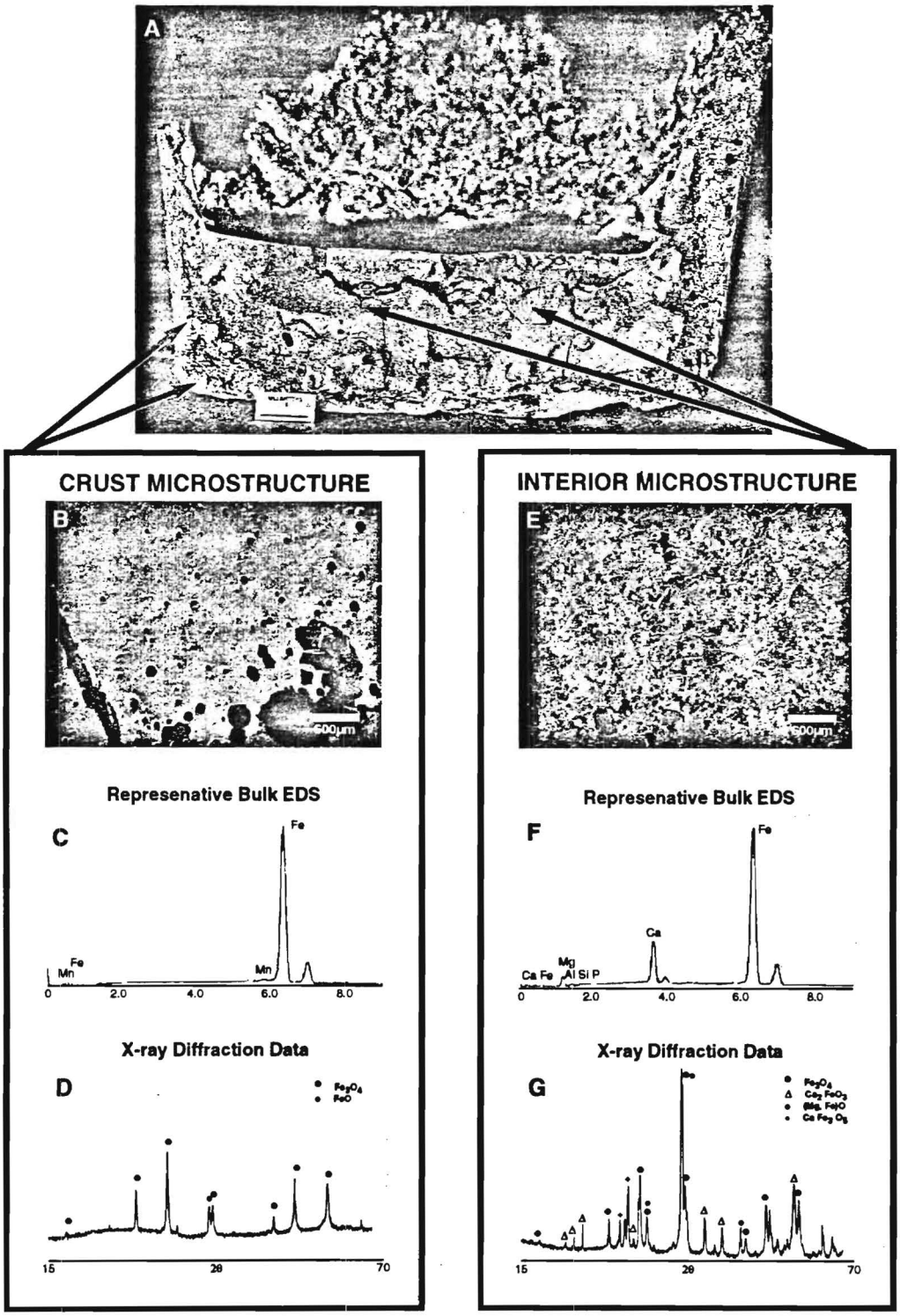


Figure 2.18. Analysis of the solidified Series 1 mixture. Item A is a cross section of the solidified product after removal from the graphite crucible. B and E are microstructure images from the crust and interior sections, respectively. Items C and F are EDS spectra for their corresponding microstructures, while D and G are the related XRD data.

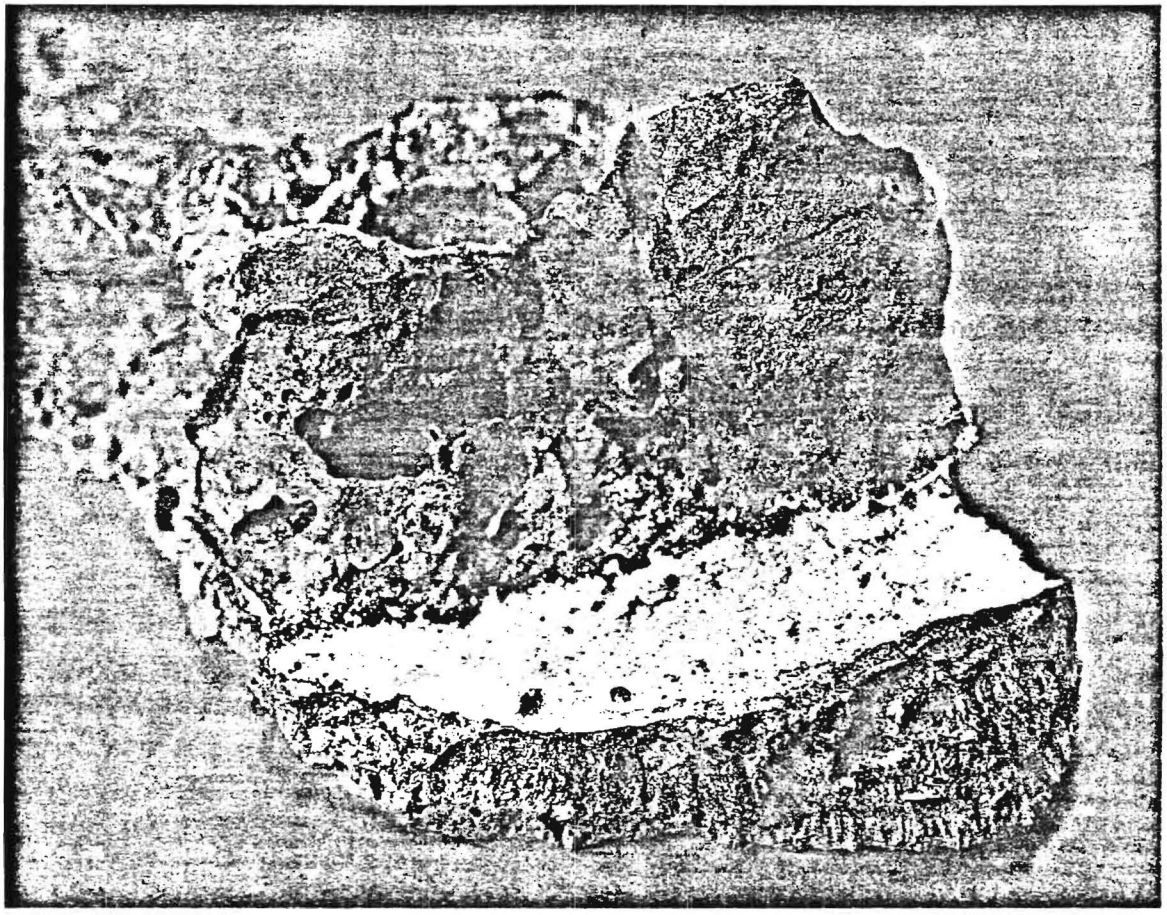


Figure 2.19. Photograph illustrating a veinlet of unreacted MgO trapped between the crust and interior regions of the solidified Series 1 surrogate.

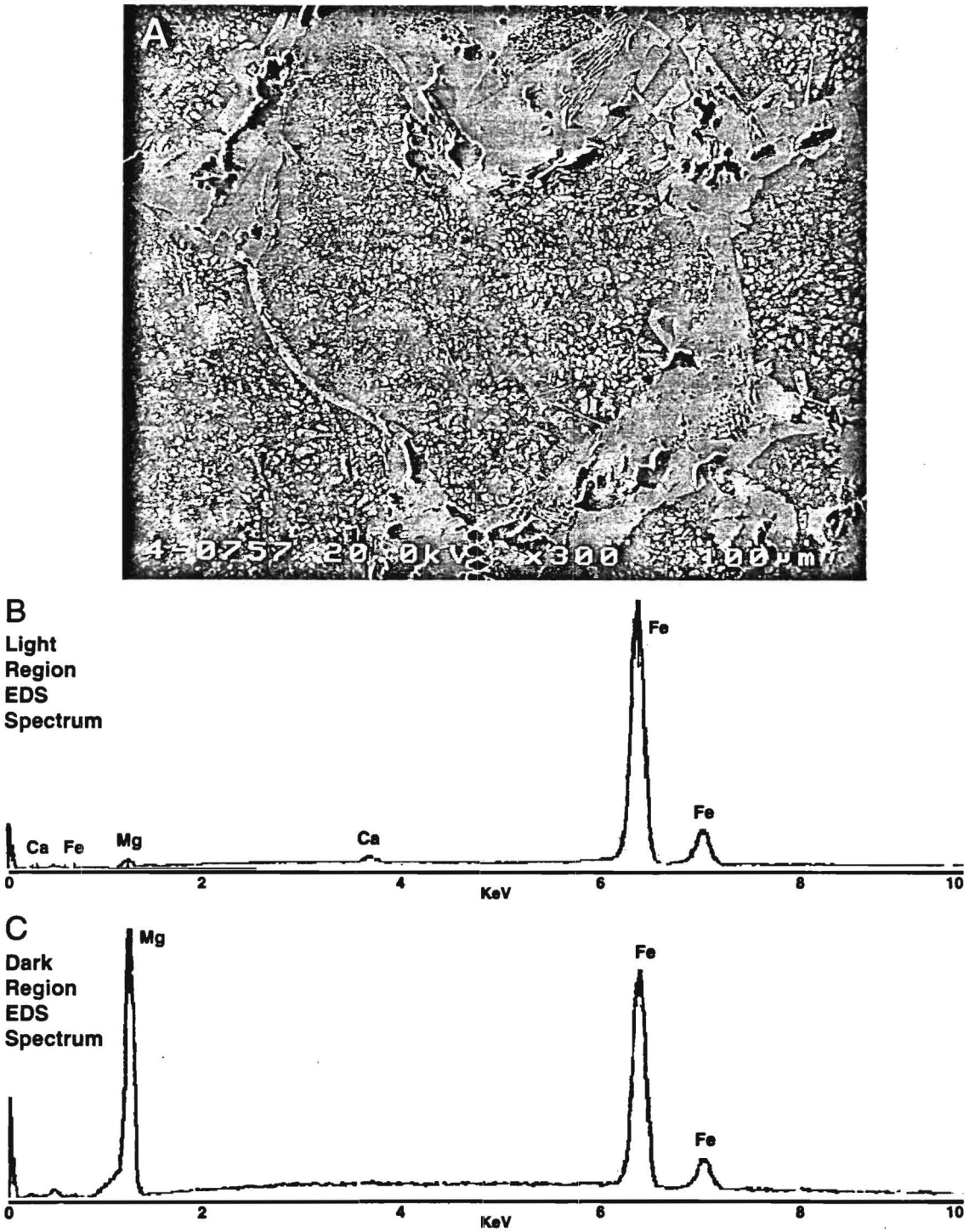


Figure 2.20. Scanning electron micrograph of the dark irregular grains seen in Figure 2.18(D) A. microstructure of dark grain at increased magnification. B and C are EDS spectra for the light grey constituent and the dark phase within the grain, respectively.

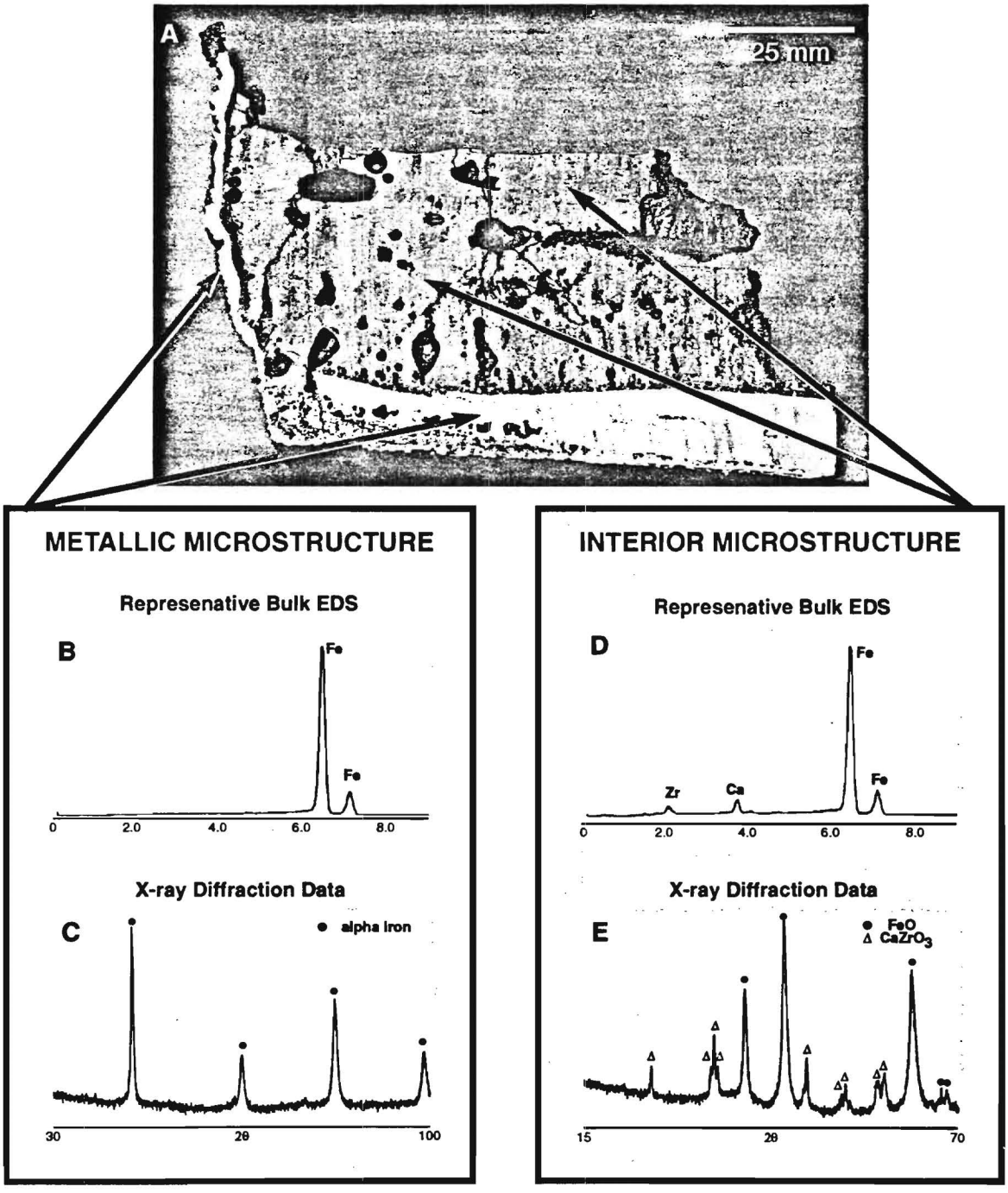


Figure 2.21. Analysis of the solidified Series 2 mixture. Item A is a cross section of the solidified product after removal from the graphite crucible. Items B and D are EDS spectra of the metallic and interior regions, respectively. Items C and E are their corresponding XRD patterns.

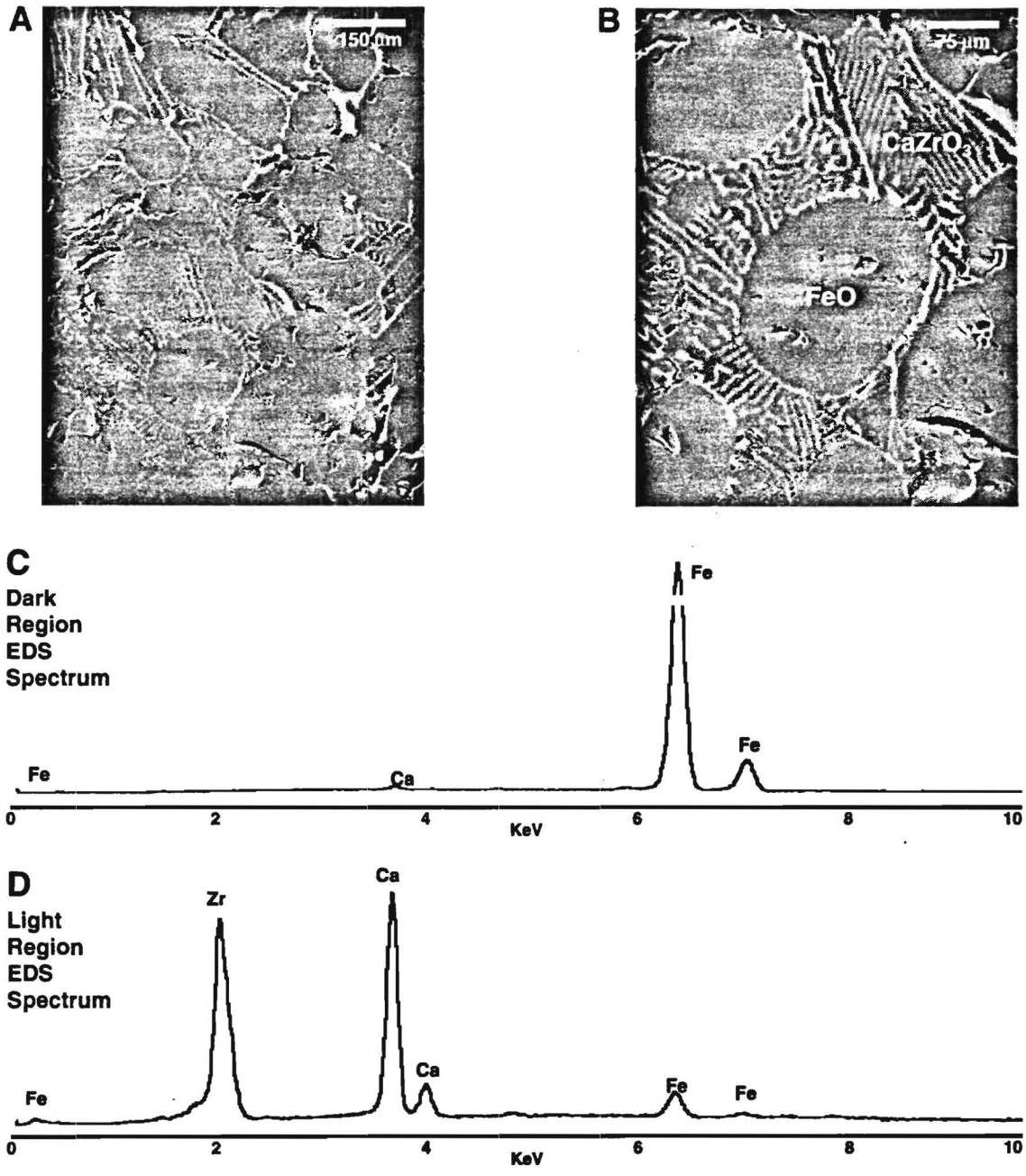


Figure 2.22. Backscattered electron images of an interior region in Figure 2.21(A and B) show the microstructure of the interior region at magnification factors of 200 and 400. C and D are EDS spectra for the darker and the lighter constituents within the microstructure, respectively.

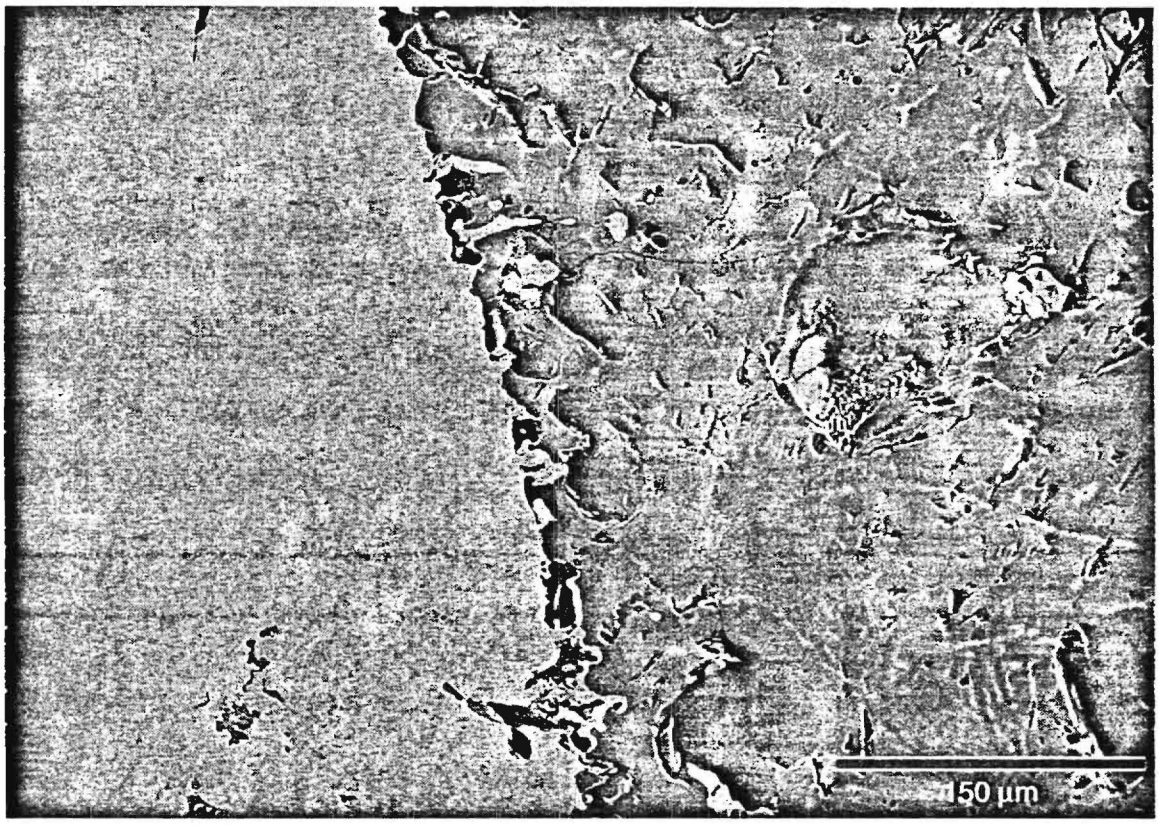


Figure 2.23. Microstructure of the interface between the iron wall (left) and the solidified interior mixture (right).

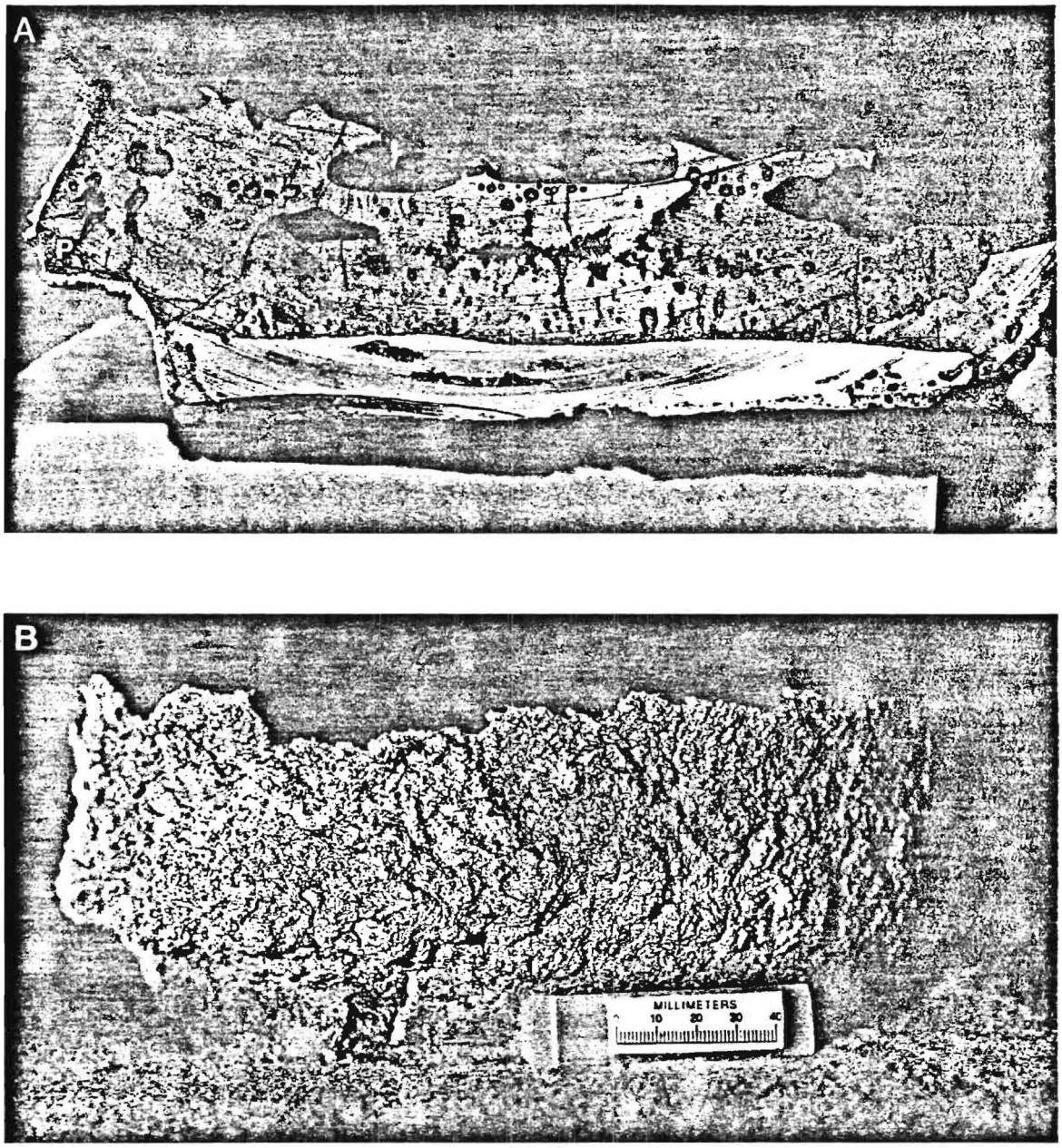


Figure 2.24. Optical micrographs showing (A) a second cross section from the solidified Series 2 melt and (B) its corresponding backside.

3. PLASMA PYROLYSIS VITRIFICATION PILOT SCALE DEMONSTRATION

Conceptual design sketches were developed to depict a process by which whole, punctured drums could be safely degassed and vitrified using a low temperature furnace in conjunction with a PPV process. Figure 3.1 summarizes the various thorium nitrate packaging configurations. Figures 3.2 and 3.3 are used in conceptualizing the low temperature/emission control and high temperature process equipment configurations. Along with mechanical material handling equipment, key process systems include:

- the low-temperature drum puncture/degas furnace;
- the PPV reactor with withdrawal systems; and
- a single off-gas treatment system common to both, with the design basis defined by the degas furnace.

The experiments conducted evaluating PPV technology for the processing of thorium surrogate materials at the Georgia Tech Plasma Application Research Facility (PARF) provide insight into the safety, hazards, and process design requirements of pilot-scale operations. Although many variables for the low-temperature process are unknown, the high-temperature surrogate processing information helps support the low-temperature process development. An objective of the PARF experiments was to identify important PPV pilot-scale process and detail design requirements.

3.1 PROCESS DESIGN REQUIREMENTS

The bench-scale PARF testing using ThO_2 surrogates generated information for the pilot-scale PPV system design and definition of lab-scale test objectives. The process design requirements are based on the surrogate work completed to date and the use of the actual thorium nitrate feed matrix for lab-scale work. Presented below is a summary listing of the data obtained to date and what will be obtained at lab scale.

3.1.1 Summary Certified Data Definition Required for Pilot-Scale PPV System Design

- Off-gas flow/composition—degas mode
  - — Total hydrocarbons (THC)—Georgia Tech
  - — Thorium nitrate NO_x generation—lab scale
  - — Air plasma generated NO, NO_2, O_2, CO, CO_2—Georgia Tech
  - — Particulate carry-over—Georgia Tech
  - — Radon transport/control—lab scale
- Bath chemistry quantification—vitrification mode for temperature, viscosity control
  - — Modifiers identified—lab scale
  - — Phase diagrams—lab scale
  - — Specific gravity profile—lab scale
  - — Fate of iron—Georgia Tech

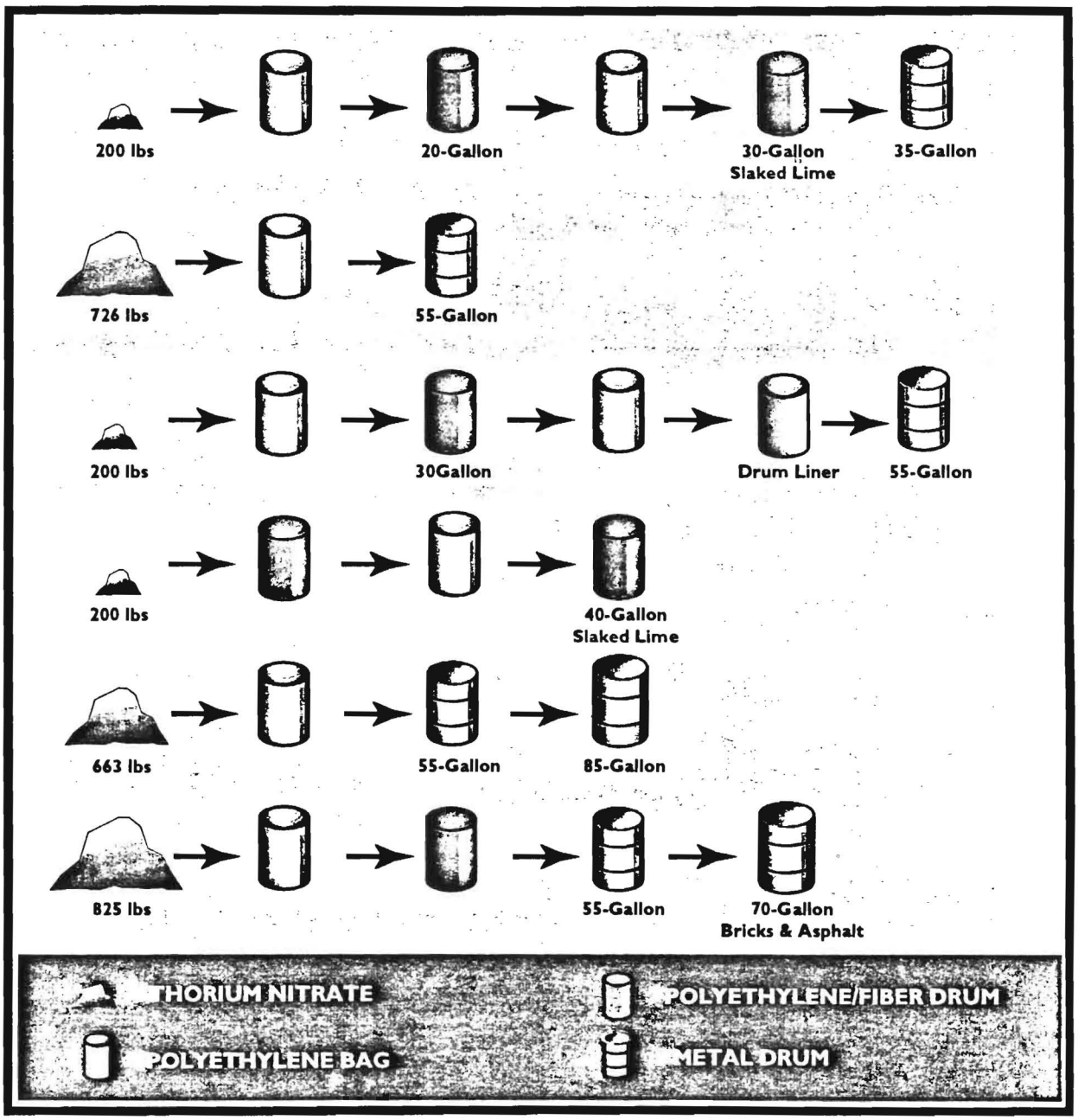


Figure 3.1. Composition of the six major drum configurations.

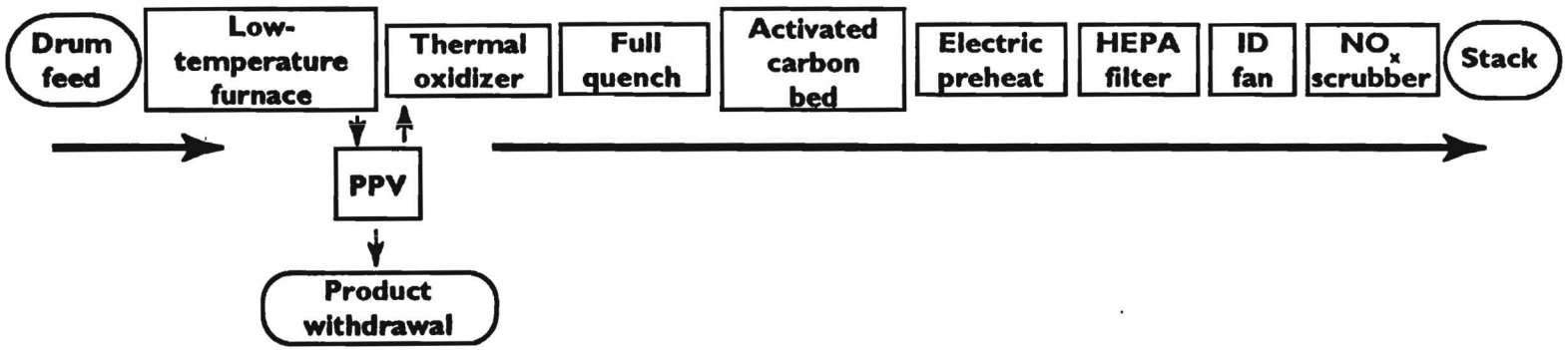


Figure 3.2 Design concept for air pollution control system.

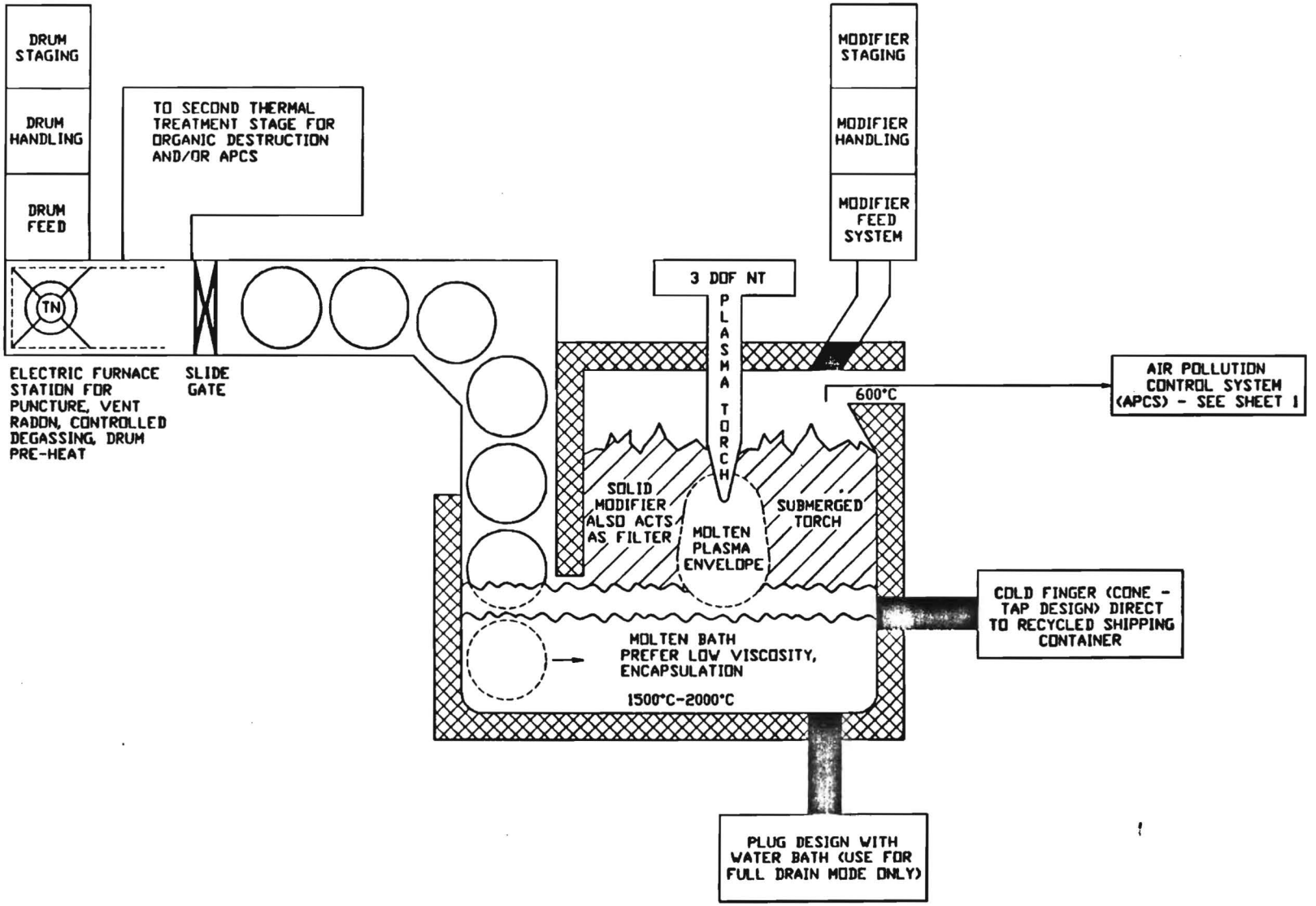


Figure 3.3. Conceptual PPV schematic.

- Chemical reactivity/explosion hazards assessment

  - Rx kinetics—lab scale
  - Heat of Rx—lab scale
  - Exotherm temperatures—lab scale
  - Gas evolution—lab scale
  - Pressure generation—lab scale

- Maryland-1 (MD-1) drum diagnostics

  - Radon control/containment for conversion—lab scale
  - Constituent separation/handling—lab scale
  - Constituent weights/composition verification—lab scale
  - Simulated package construction—lab scale
  - Drum shipments/storage, opening, venting, material transfer to Oak Ridge—lab-scale time frame
  - Nondestructive Analysis (NDA)/ultrasonics—lab-scale time frame

3.1.2 Key Lab-Scale System Test Objectives

In addition to the basic safety and process information that must be obtained at lab scale (see Section 3.2), the bench-scale PARF surrogate testing results lead to the following lab-scale, low-temperature thorium nitrate feed test objectives.

1. Dehydration/denitration must be controlled to produce an agglomerated, dense mass versus a finely divided powder product.

2. Tailoring the product of the low-temperature stage to aid in melt formation may be advantageous. Additives are required to form the desired melt characteristics. An evaluation of additives such as boric acid, SiO_2, Na_2SiO_3 shall be completed for vitrification options.

3. Immersion of the drum (and/or its contents) in the bath, along with bath chemistry, needs to be demonstrated at lab scale should the vitrification path be chosen.

4. The low-temperature thorium nitrate degas testing should be completed with results validated (with Georgia Tech results) prior to lab-scale crucible melt tests and pilot-scale PPV detail design and procurement. The results should be periodically presented in conjunction with a logic diagram that encompasses the entire scope of the thorium nitrate storage problem.

3.1.3 Key Pilot-Scale PPV System Design Requirements

1. The presence of iron offers no benefit for this chemistry with significant drawbacks; the trade-off with increased pretreatment complexity must be defined and iron removal (outer drum) is required.

2. Modifier addition is required to reduce bath operating temperature.

3. Intimate mixing of ThO_2 with modifier is required for dissolution.

4. The plasma jet must not come into direct contact with the ThO_2 (to minimize carry over).

5. Drum penetration under the bath surface is required for the in-drum feed case. Lab-scale testing is required to validate mass transfer.

6. Removal of the Al_2O_3 bricks is required.

7. The materials of construction must be controlled by rigorous specification to prevent graphite corrosion and erosion.

8. Gas phase organic destruction downstream of PPV may be required, depending on the degree of organic removal in pretreatment.

9. The PPV design should allow for a rigorous analytical thermal model, minimization of rear electrode wear (or eliminating completely), enhancement of heat transfer/energy usage efficiency, and maximization of the PPV system.

10. Specify 3 degrees of freedom torch movement (as planned) and use automated controls.

11. Specify automatic controls for controlling carrier gas pressure with flow monitoring/recording.

12. Specify modern control and data collection system by modeling after the best industry equipment and control systems in which all electrical and process (i.e., flow, temperature, pressure) parameters are monitored, alarmed, and interlocked per best practice using computers, Programmable Logic Controllers, and a distributive control and data acquisition system with graphical operator interface.

13. Specify water-cooled, jacketed PPV reactor and camera system.

14. Determine by engineering analysis the optimum plasma carrier gas (i.e., Ar, O_2, O_2/Ar, air mix) to reduce or eliminate thermal NO_x load and supply optimum environment for conversions.

15. Specify fusion promoters required for design mass/energy balance to obtain operating bath temperature (1300–1900°C) with low enough bath viscosity to allow for periodic batch removal of melt.

16. Validation of the product withdrawal function at the pilot scale is required.

17. Consideration should be given to expanding the scope of the pilot demonstration to incorporate a second waste stream (i.e., asbestos) to take advantage of the formation of a lower temperature eutectic mixture in conjunction with the submerged torch design.

18. Temperature measurement using state-of-the-art equipment and methodologies should be implemented.

19. The exact packaging of the DLA drums must be verified for mechanical design and process chemistry control.

20. Control of the operation/turbulence to minimize downstream mass transport is required, as are provisions to minimize the percent of unmelted to partially melted solid feed left as residual solid in primary chamber (hysteresis).

3.2 LAB-SCALE TESTING

Preliminary studies of surrogate nitrates (magnesium nitrate and calcium nitrate) have shown that heating rate and interaction with polyethylene can produce undesirable operating conditions (D. Laird, Science Ventures, Inc., personal communication to S. L. Camacho, Plasma Technology Corp., September 15, 1995). The following principle objectives and assessment methodologies should be implemented during feasibility studies to bracket the safety, hazard, and process design issues for the denitration and dehydration of the thorium nitrate.

- Does the nitrate-organic mixture react violently?

- Demonstrate the safe operating window (compositional, thermal, temporal).

- Does the mixture "froth" or expand upon treatment? Does it expand beyond its primary containment?

- Does the thoria (ThO_2) produce an airborne hazard? What is the size and extent of thoria dust upon treatment and handling?

- Determine off-gas characteristics (composition, volume, carry-over, radon mass transfer characteristics as a function of process conditions (i.e., operating temperature, heat rate, etc.).

These criteria will be determined through a series of small-scale evaluations of various mixtures of the drum components. From these studies the safe compositional and operating windows will be determined. Information pertaining to the off-gas composition and volume will also be obtained.

A summary of the key safety and process considerations associated with plasma processing/vitrification is exhibited as Figure 3.4. Both viscosity lab studies will not be

completed unless degas testing is successful. The basic degas testing furnace (plexiglass for viewing) is shown in Figure 3.6.

Low-temperature furnaces (0–600°C) and resistance heating methods will be used to evaluate the transformation properties [$Th(NO_3)_4 \cdot 4H_2O \rightarrow ThO_2 + NO_x + H_2O$] of the thorium nitrate. The gross safety and process parameters (e.g., exothermic nature, frothing, splattering, transport of ThO_2) of this transformation and how they are effected by the other components (calcium hydroxide, polyethylene) present in the drum will be evaluated. A general outline/overview of evaluation tasks is described below. Safety documentation and detailed procedures have been completed.

- Video monitoring and documentation of experiments.

- Use of a plexiglass enclosure to allow direct observation of crucibles being heated by direct resistance heating methods. Video collection would include crucible, time and temperature readouts, and verbal description.

- Process a small amount (milligram quantities) of individual materials as a function of temperature/time profile. Initial projected evaluation window ranges from 5°C/min (~115-minute process time) to 200°C/min (~3-minute process time).

- Process a small amount (milligram quantities) of binary mixtures as a function of temperature/time profile. Initial projected evaluation window ranges from 5°C/min (~115-minute process time) to 200°C/min (~3-minute process time). The binary mixtures would include thorium nitrate + $Ca(OH)_2$; thorium nitrate + polyethylene; $Ca(OH)_2$ + polyethylene.

- Use the information obtained from the direct observation, small-scale experiments to perform medium scale (0.5–1.0 kg) processing of "composite" drums at various thermal profiles using a low-temperature furnace.

- Evaluate the extent of materials distribution with respect to the "drum" and the nature of the thoria (ThO_2) produced.

It is relevant to briefly describe the advanced denitration/dehydration process testing, to simulate drum configurations. Furnace (0–600°C) heating with an off-gas collection system will be used to evaluate the off-gas and particulate transport properties during the denitration/ dehydration process. The experimental system will be designed for 3 scfm off-gas to accommodate use of a readily available stack sampling train for complete constituent collection and analysis. Composite drum mixtures will be used to assess the burden on the off-gas system for expected temperature/time process parameters. This information will provide input into design criteria and safe and effective operations. Composite drum design and experimental details will be developed based upon the findings and lessons learned from the basic crucible studies. Efforts are currently under way to locate a surplus furnace suitable for this canister degas application.

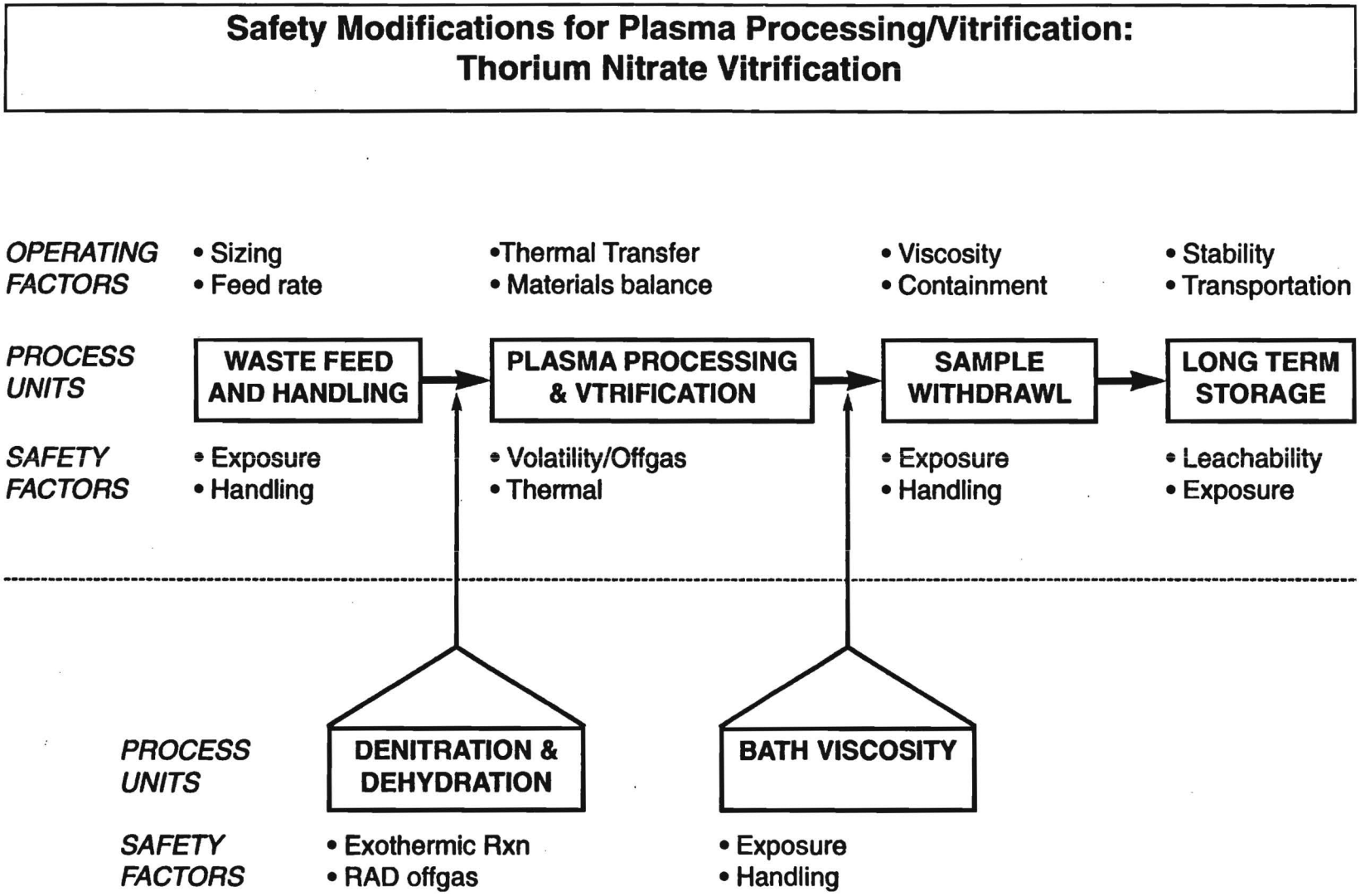


Figure 3.4. Key safety and process design considerations.

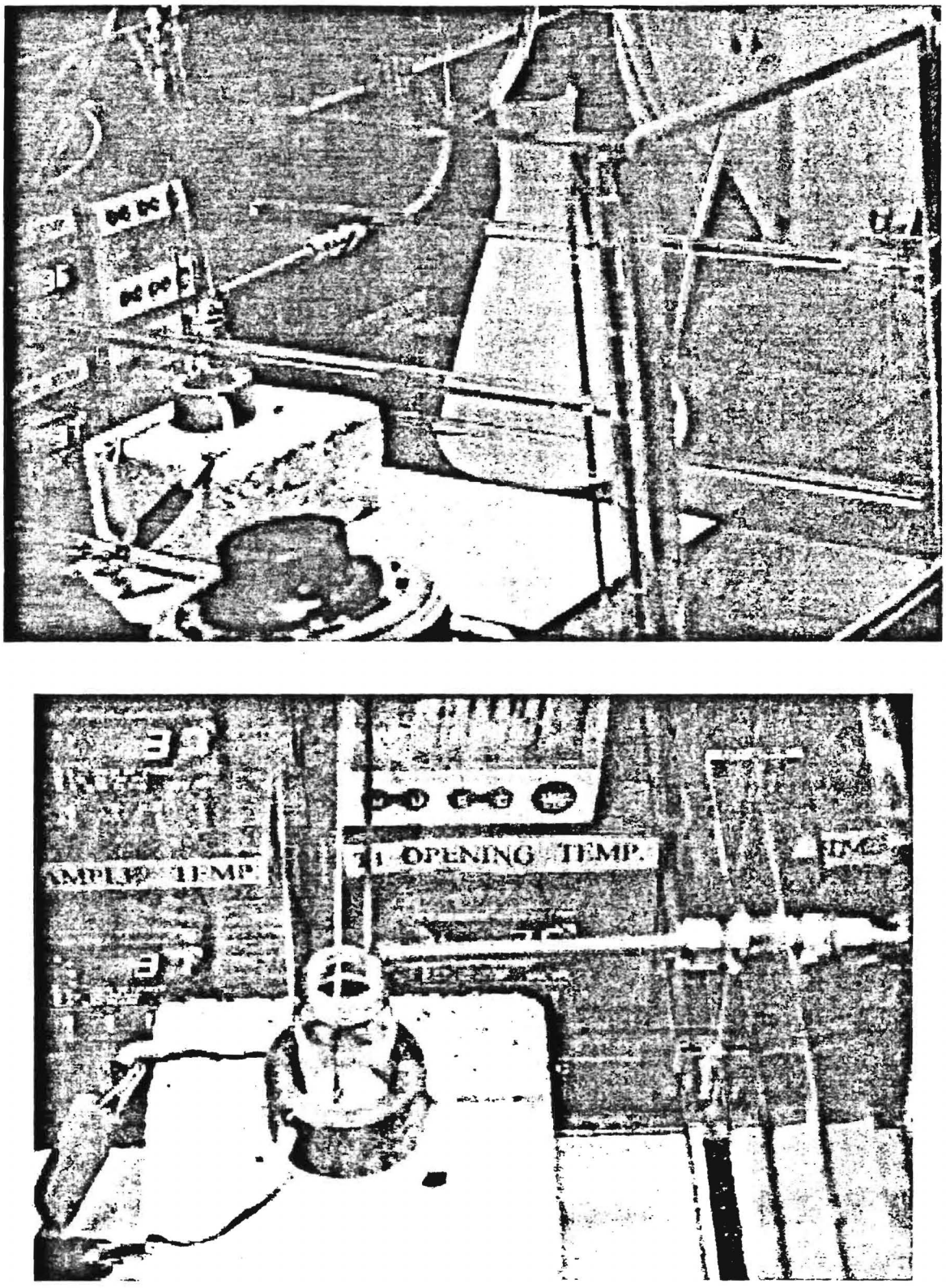


Figure 3.5. Two views of the experimental degas furnace at the K-25 Site's Materials and Chemistry Laboratory.

4. REFERENCES

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APPENDIX A

DEFENSE LOGISTICS AGENCY

THORIUM NITRATE PROGRAM

A. DEFENSE LOGISTICS AGENCY THORIUM NITRATE PROGRAM

A.1 INVENTORY

The DNSC currently stores 2596 short tons (1814 metric tons) of thorium nitrate in the Curtis Bay, Maryland facility and 952 short tons/864 metric tons in the Hammond, Indiana facility. The thorium nitrate is stored in variously sized drums with various internal configurations. The specific sequence of constituents within each drummed configuration is represented in Table A.1. The average drummed constituents and number of drums for each configuration is represented in Table A.2. The drum identification (ID) designates storage location (MD = Maryland, IN = Indiana).

A.2 DEMONSTRATION

The program is structured to evaluate the safety, hazards, and process design criteria in transforming the thorium nitrate to a low volume, stable final form. To address the considerations of thoria dust formation and the potential for violent exothermic reactions, the process design basis compomises a two stage process: low-temperature (600°C/873°K) denitration/dehydration followed by high-temperature plasma vitrification.

A drum-scale demonstration is planned for completion at the Oak Ridge, Tennessee, K-25 Site in 1996. A building at the K-25 site has been prepared to house this demonstration. The expected criteria for evaluating the acceptability of this plasma pyrolysis vitrification process are the following.

- Both volume and weight are significantly reduced.
- Radon and radioactive constituents are controllable and accountable.
- Whole drums can be processed to accomplish the sequence of dehydration, denitration, and vitrification.
- Leachability of the vitrified oxide (final product) is suitable for safe, long-term storage.
- The process meets environmental health and safety standards.

The low-temperature stage will remove the nitrate and water from the system and convert the thorium nitrate hydrates to thoria (ThO_2). The low temperature stage should also convert the calcium hydroxide to calcium oxide and the polyethylene (or asphalt) to carbon (char) and carbon oxides. The iron drum and the alumina are not expected to be greatly affected by the low-temperature process. The results of the drum-scale demonstration will be used to ascertain the need to degas the drum matrix in a vessel separate from the plasma pyrolysis vitrification (PPV) reactor.

Table A.1. DNSC thorium nitrate packaging configurations

Curtis Bay, Maryland	Package 1	Thorium nitrate inside a polyethylene bag, inside a 20-gallon fiber drum, inside a 10 mil polyethylene bag, slaked lime inside a 30-gallon cross linked polyethylene drum, inside a 35-gallon metal drum
		Thorium nitrate inventory—15,701 drums; 1,570 short tons (1,424 metric tons)
	Package 2	Thorium nitrate in a polyethylene bag inside a 55-gallon metal drum
		Thorium nitrate inventory—2,597 drums; 910 short tons (825 metric tons)
	Package 3	Thorium nitrate inside a polyethylene bag, inside a 30-gallon painted drum, inside a 10-mil polyethylene bag, slaked lime inside a cross linked polyethylene drum liner, inside a 55-gallon metal drum painted with an epoxy/phenolic resin (.6–.7-mil thickness)
		Thorium nitrate inventory—184 drums; 18 short tons (16 metric tons)
	Package 4	Thorium nitrate inside a fiber board drum, inside a 10-mil polyethylene bag, slaked lime inside a 40-gallon crossed linked polyethylene drum. The drum lid is boltedsand sealed against moisture using a polybutyllen butyl glazing tape.
		Thorium nitrate inventory—753 drums; 75 short tons (68 metric tons)
	Package 5	Thorium nitrate inside a polyethylene bag, inside a 55-gallon drum; slaked lime inside an 85-gallon painted metal drum.
		Thorium nitrate inventory —66 drums; 22 short tons (20 metric tons)
Hammond, Indiana	Package 1	Thorium nitrate inside a polyethylene bag, inside a fiber drum, inside a 55 gallon drum, vermiculite inside a 70 gallon painted metal drum Thorium nitrate inventory - 2,308 drums - 952 short tons (864 metric tons)

Note: There are 4 bricks and about 3 inches of asphalt in the bottom of the 70-gallon overpack to support the 55-gallon inserted drum. The void space between the drums is filled with vermiculite or similar material

Table A.2. Thorium nitrate storage drum configuration*

Item	MD-1	MD-2	MD-3	MD-4	MD-5	IN-1
	Amount of components in drums (pounds**) (1 Kg = 2.2 lb)					
$Th(NO_3)_4*4H_2O$	200	726	200	200	663	825
$Ca(OH)_2$	44	0	44	105	269	0
Polyethylene	30	1	39	34	1	22
Fe	48	60	60	0	152	139
Al_2O_3	0	0	0	0	0	63
Asphalt	0	0	0	0	0	43
Drum Size (Liter)	132.5	208.2	208.2	151.4	321.7	265
Drum Size (gallon)	35	55	55	40	85	70
Number of Drums	15,701	2,597	184	753	66	2308

*The drum designations indicate storage locations (MD—Maryland; IN—Indiana).

**One kilogram is equivalent to 2 pounds.

The residuals in the drum from the low-temperature process (e.g., Fe, ThO_2, CaO, C, Al_2O_3) will then be processed by plasma pyrolysis vitrification (PPV). The objective of the high-temperature stage is to maximize the density of the final product and to help encapsulate and/or integrate the thoria into a glass or slag matrix.

A.2.1 Special Process Design Considerations

The thorium in its present configuration will have significant radon (^{220}Rn) gas collected within the crystalline structure. The radon gas is from the ^{232}Th decay. The radon gas will be released as the drum is vented and the thorium is heated. ^{220}Rn will be collected in the air pollution control (APC) carbon adsorption unit, HEPA filters/aqueous scrubber stream. Thorium nitrate hydrates have a variable water content, cake very badly, and are difficult to meter or transfer as a solid. Transfers of whole, unopened drums were adopted for the baseline process design to minimize pretreatment/handling requirement and take full advantage of the high temperature plasma heating process.

A.2.2 Activity Level Basis

Naturally occurring thorium is almost entirely ^{232}Th plus 1.35E-8 wt % ^{228}Th.[1] In irradiated thorium, the ^{228}Th would be higher due to decay of ^{232}U. The French, India, and U.S. sources are all 1.3E-8 wt % ^{228}Th. ^{230}Th levels depend on how much uranium was not separated when the material was prepared. The French material is about 14 ppm U by wt. The India and U.S. material are below 1 ppm U. This trend seems to be properly reflected in ^{230}Th levels of each source.

Activity of 1.0g of $^{232}Th = 1.1 \times 10^2$ nCi/g (verified with the DOT charts)
Activity of 1.0g of $ThO_2 = 1.1 \times 10^2 \times 232/264$—97 nCi/g
Activity of 1.0g of $Th(NO_3) \cdot 4\ H_2O = 1.1 \times 10^3 \times 232/552 = 46$ nCi/g
Activity of 1.0g of $ThO_2 = 970$ nCi/g (in equilibrium with 10 daughters)
Activity of 1.0g of $Th(NO_3)_4 \cdot 4\ H_2O = 460$ nCi/g (in equilibrium with 10 daughters)

Since the ^{220}Rn daughter activity is in equilibrium with ^{232}Th, its activity will be the same as ^{232}Th. Therefore, if all the ^{220}Rn were released and captured in the HEPA filters, the activity of the filters would be 97 nCi/g ThO_2. The ^{220}Rn half-life is very short (56 sec). The radioactivity from other radioactive isotopes, such as ^{226}Ra from ^{228}Th, may be caught in the filters. The average radiological readings at drum contact is 5.5 MR/hr.

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[1]Reference for 1.35 E-8 wt % ^{228}Th in natural thorium and ^{230}Th being associated with natural uranium is M. Benedict, T.H. Pickford, H.W. Levi, Nuclear Chemical Engineering, Second Edition, McGraw-Hill Book Company, New York, NY, 1981, pp. 284–285.

A.2.3 Radiological Considerations—General Discussions

The storage, handling, processing and transport of thorium (and associated radioactive progeny) will require radiological exposure monitoring as well as administrative and engineering controls to maintain exposures below regulatory limits and to levels that are as low as reasonably achievable (ALARA). Properly packaged, thorium nitrate compounds present only an external radiation hazard. However, associated with vitrification, thorium and radioactive progeny exposures are possible with products in both contained and uncontained forms. Further, nonradioactive elements which serve to attenuate radiation in thorium nitrate may be liberated or added and radiation levels from comparable masses of pre- and post-vitrification thorium compounds may differ. The demonstration will provide information necessary to develop a vitrification specific radiological protection program and a basis for comparison with radiological protection programs of alternative thorium nitrate treatment processes.

A.2.4 Naturally Occurring Radioactive Materials

There are three naturally occurring radioactive decay series which may be found in and extracted from ores. They are denominated as the uranium series, the thorium series, and the actinium series. The thorium series is headed by the very long lived ^{232}Th. The series begins with ^{232}Th that decays to radionuclides of radium, actinium, thorium, radon (a mobile, inert gas), polonium, lead, bismuth, thallium and ends with transformation to stable (i.e., nonradioactive) lead. In the absence of physical separation processes, an equilibrium is reached in which the number of atoms of each nuclide of the radioactive series that decays during a specific time interval nearly equals the number of decays of the parent nuclide in the series. The demonstration will provide information regarding the state of equilibrium in the series and the affects on that equilibrium by the vitrification process.

A.2.5 Characteristics Related to Radiation Dose

The potential radiation dose that workers or the public may receive from exposure to radioactive material (i.e., the thorium decay series) is determined by a number of factors. These include the amount of material involved, the types of radiation emitted by the material involved, the chemical and physical form of the material, the solubility of the material, the particle size distribution of the material, the duration of the exposure, the amount of material that may be resuspended from past releases (and on-going operations), dispersion and dilution conditions at the time of exposure, the ingestion pathways involving contaminated water, food stuffs and animal feeds, and the demographic and physiological characteristics of the population exposed. The demonstration will provide information regarding these factors relative to the thorium nitrate vitrification process.

A.2.6 External Radiation

External sources of radiation exposure to workers are caused by the concentrations and quantities of naturally occurring radioactive material present, especially the concentrations and quantities of nuclides such as actinium, lead, bismuth and thallium which are intermediate in the thorium series. External radiation levels from thorium nitrate in storage varies. A sampling indicates that one storage configuration, an isolated drum, yields contact exposure levels of approximately 5 mR/hr, one foot levels of approximately 2 mR/hr, and three foot levels of approximately 1 mR/hr. During vitrification chemical and physical reactions can result in changes in the relative concentrations and quantities of the naturally occurring radioactive materials as well as the nonradioactive constituents which provide attenuation of radiation. Further, piping and process vessels, product containers and waste/filtration systems will have various concentrations and quantities of naturally occurring radioactivity. The demonstration process will provide information regarding the distribution of naturally occurring radioactive material and resultant radiation fields associated with thorium nitrate vitrification.

A.2.7 Airborne Radioactivity

Airborne radioactivity may occur during the vitrification process. Airborne radionuclides will include thorium decay series radionuclides as resuspended particulates and radon-220 gas. The concentrations may vary widely. There may be many situations, such as grinding and crushing, dry feed transfer, off-gassing, sampling and analytical procedures, maintenance, packaging, waste disposal and accidents that result in exposure to airborne radioactivity. Dust particles may contain alpha particle, beta particle and gamma photon emitting radionuclides which can irradiate internal organs of the body after inhalation or ingestion. The demonstration will provide information regarding the concentrations and control of airborne radioactivity associated with thorium nitrate vitrification.

A.2.8 Surface Contamination

Transferable surface contamination can be a source of inhalation and ingestion of radioactive materials. Therefore, control of surface contamination is an essential component of any radiological protection program. Operations that may result in contaminated surfaces include crushing and grinding, process equipment maintenance, agitation, and decontamination. The demonstration process will provide information regarding the control and monitoring of surface contamination associated with thorium nitrate vitrification.

Much of the above information is applied from industry recommendations:
NCRP (1993). National Council on Radiation Protection and Measurements. *Radiation Protection in the Mineral Extraction Industry*, NCRP Report No.118 (National Council on Radiation Protection and Measurements, Bethesda, Maryland).

A.3 THERMAL DATA ON DRUM CONSTITUENTS

A.3.1 Thorium Nitrate Hydrates

Data in the "Gmelin Handbook of Inorganic Chemistry" lists tetrahydrate, pentahydrate and hexahydrates of thorium nitrate as solid phases. One phase study of the $Th(NO_3)_4 \cdot XH_2O$ system revealed the melting temperatures of the tetrahydrate and hexahydrate at ~150°C (423°K) and ~110°C (383°K), respectively.

Thermogravemetric (TGA) data for $Th(NO_3)_4 \cdot 5\ H_2O$ heated in air at 6°C/min shows the final temperature of decomposition to be between 400–500°C (letter memo comments on the Decomposition Behavior of Thorium Nitrate Hydrates, P.A. Haas to W.H. Hermes, 11/11/94). The T.G.A. shows a change of slope occurring before ~200°C (473°K) with initial weight losses probably as water and possibly corresponding to the formation of such hydrates as $Th(NO_3)_4 \cdot 3\ H_2O$ in air which reportedly forms between 118–144°C on heating $Th(NO_3)_4 \cdot 4\ H_2O$ in air. Following the change of slope there appears to be a constant weight loss up to ~300°C (573°K) where the curve flattens which could correspond to the phase $ThO(NO_3)_2 \cdot 0.5\ H_2O$. It should be noted that it is well documented that increased heating rates shift reaction temperatures to lower values. Rapid heating of either surrogate or real drums will affect reaction temperatures and kinetics.

Based on a literature search completed, a series of steps that occur on heating hydrated thorium nitrate directly in a plasma torch, were postulated. The reaction steps are as follows:

1. The solid melts to yield a solution of medium viscosity.

2. The solution becomes more viscous with the loss of water but with minimal loss of nitrates.

3. The losses of nitrate and water converts the material to a very thick melt and then to a slag or glass.

4. Continued change sees the solid becoming a porous ThO_2 agglomerate with a relatively low bulk density, perhaps as low as only 20% of theoretical density.

5. The agglomerates have been shown to be in the range of 20–30 microns but may contain even smaller thoria crystallites. Attempts to form dense thoria by thermal denitration were aided by small additions of Al_2O_3 or CaO. This latter compound will be present in much larger amounts as a constituent in the drum.

A.3.2 $Ca(OH)_2$

$Ca(OH)_2$ supposedly interfaces with the thorium nitrate separated by a plastic surface in several of the drum configurations. Literature values for the thermal decomposition of $Ca(OH)_2$ are listed as ~600°C (873°K). However, possibilities exist for at least partial conversion of $Ca(OH)_2$ to probably $CaCO_3$ which has a substantially higher temperature of melting. In addition,

it is known that ThO_2 + CaO can react to form $ThCaO_3$ which melts at 2300°C (2573°K). Some reaction of these phases at the interface might be expected.

A.3.3 Iron

Drums that hold the mixture are most probably a low carbon steel. Years of exposure to ambient conditions have affected the exterior surface. Oxidation of the surface is probably minimal because of exterior paint nevertheless we need to recognize that possibility. The presence of iron oxides would not change the melting temperatures in a significant way since metal and oxides form melts at ≤1550°C (1823°K). An important question is how iron will act in a molten bath with other metal oxides, e.g., CaO, ThO_2 and possible glass formers.

A.3.4 Additives

Since ThO_2 is the most refractory oxide, it is envisioned that additives may be required to lower the fusion temperature of the molten bath. The formulation of a glass forming mixture should take advantage of the existing elements present (Ca, Fe). The minimum design basis temperature at which the bath should be maintained is selected to be 1550°C based on iron oxides and iron melting at near 1500°C (1773°K). One phase diagram reference (Nuclear Technology, T.S. Snidhar, 1985) showed the liquidus temperature for B_2O_3—ThO_2 can be as low as 1500°C with 30 to 90 mole% of ThO_2 present in the system. The use of additives will be investigated to minimize bath temperature and control viscosity.

An alternate use of additives may be to suppress entrainment of thoria dust by enhancing adhesion and agglomeration of micron-size ThO_2 crystals. Such an additive could be introduced in minor proportion before low temperature thermal denitration. It could reduce filter burden in both low and high temperature process steps. For example, boron forms an oxide eutectic with thorium melting at only 350°C (623°K). The eutectic forms before decomposition of thorium nitrate is complete, so that thoria particles are never free of the binding influence of liquids.

APPENDIX B

DEFENSE LOGISTICS AGENCY

DRUM CONSTITUENT SUMMARY

Table B.1. Assumptions for weights of drum constituents

Drum Type	Weight (lb)	Barrier material	Compound	Weight percent
1/Md	—	—	—	
[44.3% inventory per attached table]	200	TN	$Th(NO_3)_4 \cdot 3\ H_2O$	62
	1	10-mil PE bag (typ)[2]	CH_2	
	12	20-ga PE drum	CH_2	4
	1	PE bag	CH_2	
	44	10-ga slaked lime[3]	$Ca(OH)_2$	14
	16	30-ga x-linked PE fiber drum	CH_2	5
	48	35-ga metal drum	Fe	15
Total	322 lb			
2/Md	726	TN	$Th(NO_3)_4 \cdot 3\ H_2O$	
[25.7% inventory]	1	PE bag	CH_2	
	60	55-ga metal drum	Fe	
Total	787 lb			
3/Md	200	TN	$Th(NO_3)_4 \cdot 4\ H_2O$	
[0.5% inventory]	1	PE bag		
	16	30-ga PE drum	CH_2	
	1	PE bag	CH_2	
	44	10-ga slaked lime	$Ca(OH)_2$	
	21	40-ga PE drum liner	CH_2	
	60	55-ga metal drum painted with epoxy/phenolic resin (.7 mil)	Fe	
Total	343 lb			
4/Md				
[2.1% inventory]	200	TN	$Th(NO_3)_4 \cdot 4\ H_2O$	
	12	20-ga fiber PE drum	CH_2	
	1	PE bag	CH_2	
	105	Slaked lime	$Ca(OH)_2$	
	21	40-ga x-linked PE drum	CH_2	
Total	339 lb			
5/Md			$Th(NO_3)_4 \cdot 4\ H_2O$	
[0.6% inventory]	663	TN	CH_2	
	1	PE bag	Fe	
	60	55-ga metal drum	$Ca(OH)_2$	
	269	Slaked lime	Fe	
	92	85-ga metal drum		
	1,085 lb			

Table B.1. Assumptions for weights of drum constituents (Cont'd)

Drum Type	Weight (lb)	Barrier material	Compound	Weight percent
1/In		TN	$Th(NO_3)_4 \cdot 4\ H_2O$	
[26.8% inventory]	825	PE bag	CH_2	
	1	40-ga PE drum	CH_2	
	21	55-ga metal drum	Fe	
	60	72-ga painted metal	Fe	
	79	drum		
		Bricks	Al_2O_3	
	63	Asphalt	CH_2	
	43			
	1,092			

(1)In several cases, drum dimension and material information was not available so assumptions were applied (see table).
(2)PE and cardboard composition per Perry's Handbook Chem Engineering.
(3)Calculated based on dimensions for available drums at 30 lb/ft^3 bulk density.

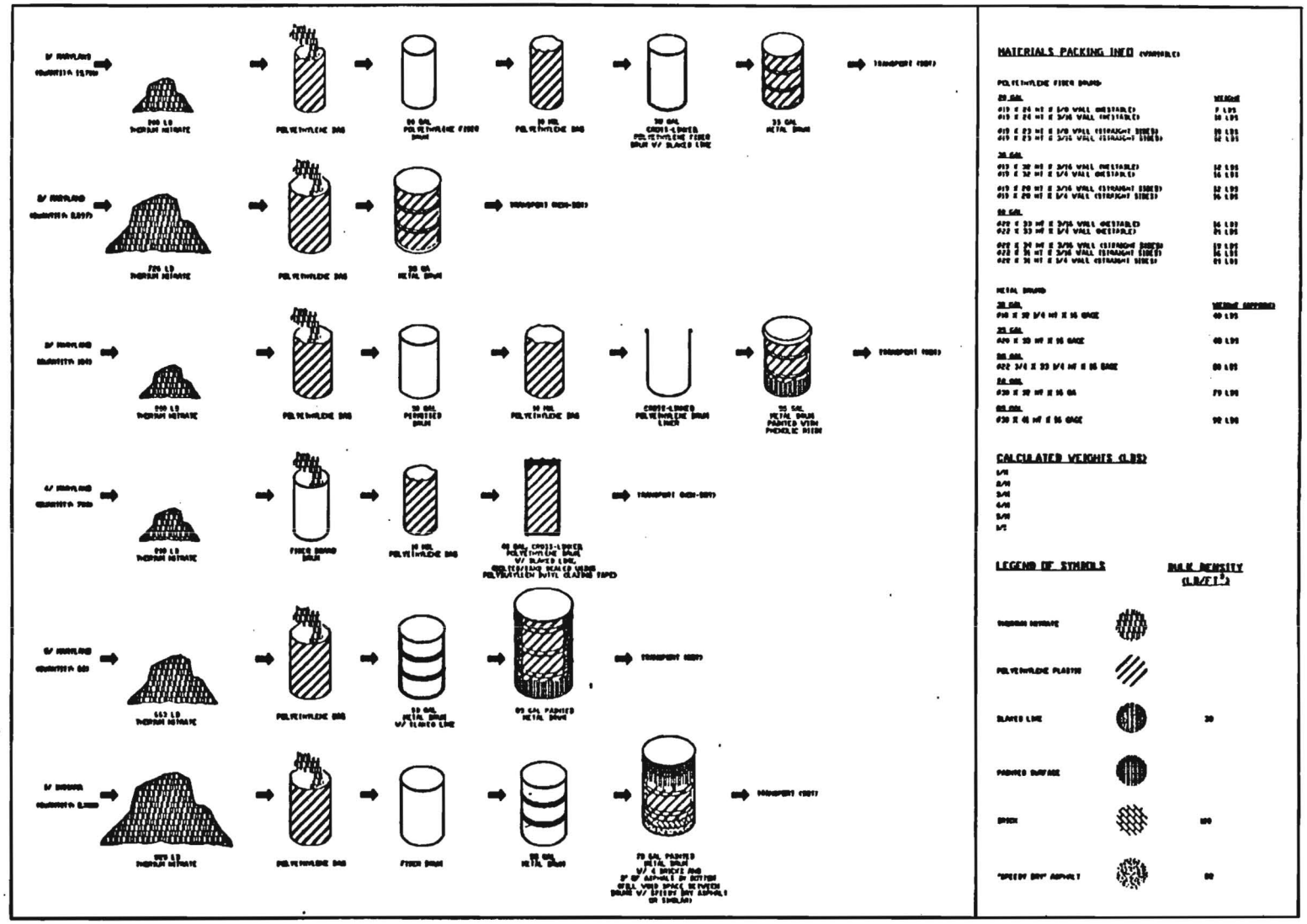


Figure B.1. Thorium nitrate packaging information summary.

Composition (lbs) of the various components in the DLA TN drums

TN = $TH(NO_3)_4 \cdot {}_4H_2O$

	MD-1	MD-2	MD-3	MD-4	MD-5	IN-1	
N	200	726	200	200	663	825	
$Ca(OH)_2$	44	0	44	105	269	0	
PE	30	1	39	34	1	22	
Fe	48	60	60	0	152	139	
Al_2O_3						63	
Asphalt						43	
Total wt	322	787	343	339	105	1092	
# Cont.	15701	2597	184	753	66	2308	
Tot wt % Inv.	50.5	20.4	0.6	2.6	0.7	25.2	
TN wt % Inv.	43.9	26.3	0.5	2.1	0.6	26.6	< DLA #s
# Drums % Inv	72.7	12.0	0.9	3.5	0.3	10.7	

The Following numbers are normalized to a 100% composition for TN+$Ca(OH)_2$+PE+Fe

Wt %	MD-1	MD-2	MD-3	MD-4	MD-5	IN-1	Avg. Comp.
TN	62.1	92.2	58.3	59.0	61.1	83.7	69.4
$Ca(OH)_2$	13.7	0.0	12.8	31.0	24.8	0.0	13.7
PE	9.3	0.1	11.4	10.0	0.1	2.2	5.5
FE	14.9	7.6	17.5	0.0	14.0	14.1	11.4

Tot wt % Inv	MD-1	MD-2	MD-3	MD-4	MD-5	IN-1	Weighted Comp
TN	31.4	18.8	0.4	1.5	0.4	19.0	71.5
$Ca(OH)_2$	6.9	0.0	0.1	0.8	0.2	0.0	7.9
PE	4.7	0.0	0.1	0.3	0.0	0.5	5.6
Fe	7.5	1.6	0.1	0.0	0.1	3.3	12.5

TN wt % Inv	MD-1	MD-2	MD-3	MD-4	MD-5	IN-1	Weighted Comp
TN	27.2	24.3	0.3	1.2	0.4	20.1	73.5
$Ca(OH)_2$	6.0	0.0	0.1	0.7	0.2	0.0	6.9
PE	4.1	0.0	0.1	0.2	0.0	0.5	4.9
Fe	6.5	2.0	0.1	0.0	0.1	3.4	12.1

#Drums % Inv	MD-1	MD-2	MD-3	MD-4	MD-5	IN-1	Weighted Comp
TN	45.1	11.1	0.5	2.1	0.2	8.1	67.0
$Ca(OH)_2$	9.9	0.0	0.1	1.1	0.1	0.0	11.2
PE	6.8	0.0	0.1	0.3	0.0	0.2	7.4
Fe	10.8	0.9	0.1	0.0	0.0	1.4	13.3

	MD-1	Wt %	Tot wt %	TN wt %	# Drums %
		**Based upon inventory Weighted Compositions			
TN	62.1	69.4	71.5	73.5	67.0
$Ca(OH)_2$	13.7	13.7	7.9	6.9	11.2
PE	9.3	5.5	5.6	4.9	7.4
Fe	14.9	11.4	12.5	12.1	13.3

Weight Distribution (in grams) for Test Capsules:
0.1lb = 45g, 0.25lb = 114g, 0.50lb = 227g, 0.75lb = 341g, 1.0lb = 454g

Based upon: MD-1

MgN (TN)	45.0	114.0	227.0	341.0	454.0
$Ca(OH)_2$	9.9	25.1	49.9	75.0	99.9
PE	6.8	17.1	34.1	51.2	68.1
Fe	10.8	27.4	54.5	81.8	109.0
Total	72.5	183.5	365.5	549.0	730.9

Based upon: Avg. Wt %

MgN (TN)	45.0	114.0	227.0	341.0	454.0
$Ca(OH)_2$	8.9	22.5	44.8	67.4	89.7
PE	3.6	9.1	18.1	27.2	36.2
Fe	7.4	18.7	37.1	55.8	74.3
Total	64.8	164.2	327.1	491.3	654.1

Based upon: Tot wt % Inv.

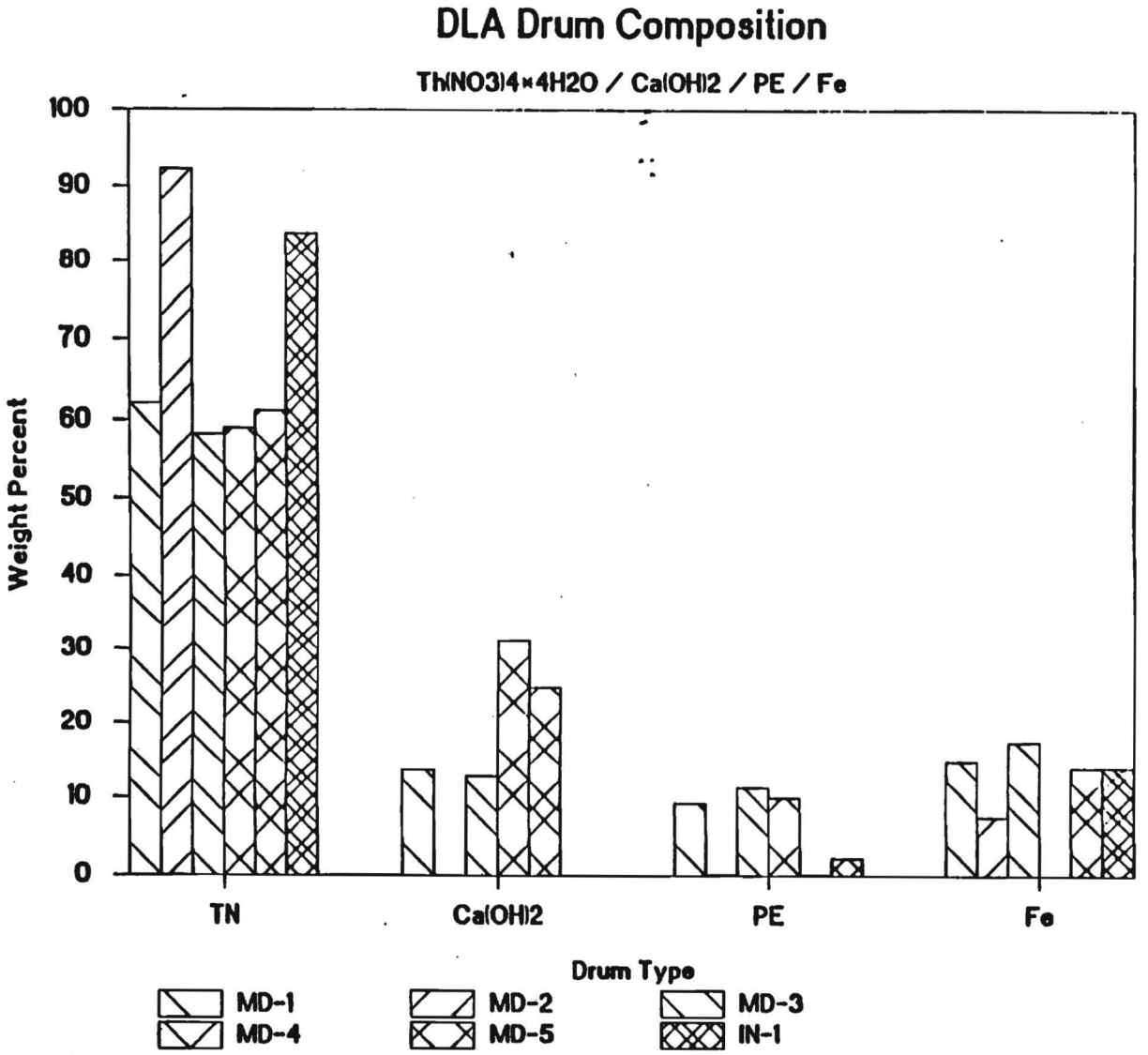
MgN (TN)	45.0	114.0	227.0	341.0	454.0
$Ca(OH)_2$	5.0	12.7	25.2	37.9	50.5
PE	3.5	8.9	17.7	26.5	35.3
Fe	7.9	19.9	39.7	59.6	79.3
Total	61.4	155.5	309.6	465.0	619.1

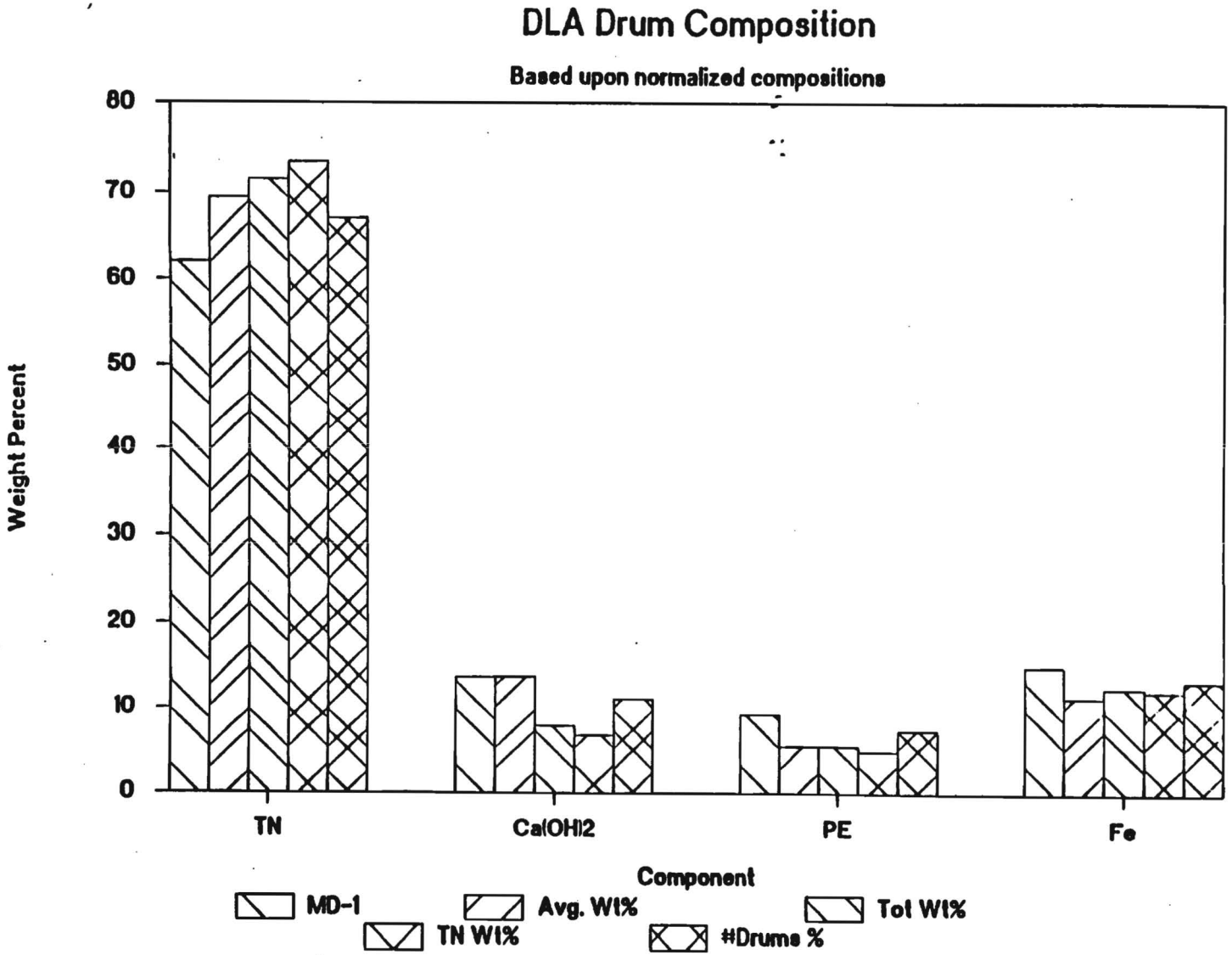
Based upon: TN Wt % Inv.

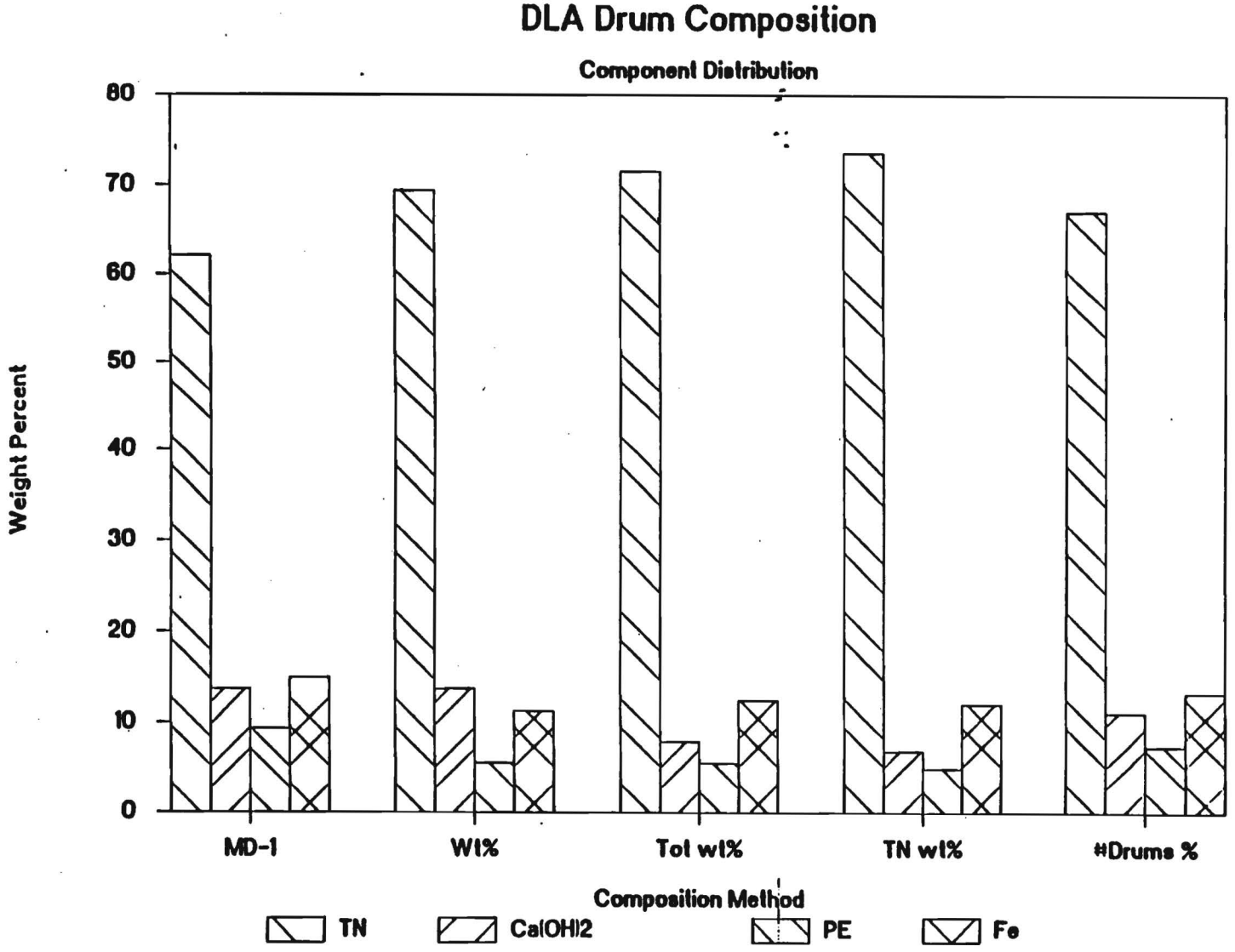
MgN (TN)	45.0	114.0	227.0	341.0	454.0
$Ca(OH)_2$	4.2	10.6	21.2	31.8	42.4
PE	3.0	7.6	15.2	22.8	30.4
Fe	7.4	18.8	37.4	56.1	74.7
Total	59.6	151.0	300.8	451.8	601.5

Based upon: # Drums % Inv.

MgN (TN)	45.0	114.0	227.0	341.0	454.0
$Ca(OH)_2$	7.5	19.0	37.9	56.9	75.8
PE	5.0	12.7	25.2	37.9	50.4
Fe	8.9	22.6	45.0	67.7	90.1
Total	66.4	168.3	335.2	503.5	670.3


DLA Drum Composition
Th(NO3)4•4H2O / Ca(OH)2 / PE / Fe
100
90
80
70
60
50
40
30
20
10
0
Weight Percent
TN
Ca(OH)2
PE
Fe
Drum Type
MD-1
MD-2
MD-3
MD-4
MD-5
IN-1


DLA Drum Composition
Based upon normalized compositions
Weight Percent
80
70
60
50
40
30
20
10
0
TN
Ca(OH)2
PE
Fe
Component
MD-1
Avg. Wt%
Tot Wt%
TN Wt%
#Drums %


DLA Drum Composition
Component Distribution
Weight Percent
80
70
60
50
40
30
20
10
0
MD-1
Wt%
Tol wt%
TN wt%
#Drums %
Composition Method
TN
Ca(OH)2
PE
Fe

APPENDIX C

ACTUAL SURROGATE FEED DATA

Fe Can with Lid #	Weights (g)								
	Digital Scale 1			Digital Scale 1		Digital Scale 2			
	Record Fe Can with Lid (g)			Record MgO (g)		Record CeO_2 (g)		Record ZrO_2 (g)	
	Can	Lid	Can + Lid	Target	Actual	Target	Actual	Target	Actual
285.10 1	73.86 +	7.41	= 81.27	201.9 ± 10	201.90	1 ± .05	1.00	1 ± .05	1.00
282.32 285.98 2	72.94 +	7.51	= 80.45	199.86 ± 10	199.86	1 ± .05	1.01	1 ± .05	1.00
286.08 3	73.90 +	7.63	= 81.53	202.55 ± 10	202.55	1 ± .05	1.00	1 ± .05	1.01
285.19 4	73.57 +	7.41	= 80.98	201.18 ± 10	202.20	1 ± .05	1.00	1 ± .05	1.01
283.56 5	73.10 +	7.77	= 80.87	200.91 ± 10	200.90	1 ± .05	1.00	1 ± .05	1.00
284.41 6	73.63 +	7.41	= 81.05	201.35 ± 10	201.35	1 ± .05	1.01	1 ± .05	1.00
279.49 7	72.28 +	7.37	= 79.65	197.85 ± 10	199.83	1 ± .05	1.01	1 ± .05	1.00
284.30 8	73.57 +	7.41	= 81.02	201.28 ± 10	201.28	1 ± .05	1.00	1 ± .05	1.00
284.64 9	73.75 +	7.37	= 81.12	201.53 ± 10	201.53	1 ± .05	1.00	1 ± .05	1.00
283.94 10 (control sample)	73.55 +	7.36	= 80.92	201.03 ± 10	201.03	1 ± .05	1.00	1 ± .05	1.00
No. wt. measurements	10				2012.43 10		10.03 10		10.02 10

Packaging Requirements
Refer to ALDRICH quote attached

Health Safety Requirements
1. Follow MSDS handling instructions
2. Wear PPE per GA Tech practice. Consult the MSDS for general guidance

Special Requirements
1. Manually shake each closed can for 30 seconds after the ZrO_2/CeO_2 have been added
2. Record weight measurements to 4 significant figures
3. Mark tare weight of can with lid and "Series 1, Run 1" on each can
4. Store cans in air conditioned environment

NOTE: Nail punch three vent holes in the top of each can will be done in field as cans are fed

Major Equipment Requirements
(A) Ventilation hood
(B) 2-digital scales (one for ~1 lb measurements, one for ~1g measurements)

Date 9/6/95
Initial GJG

SURROGATE PREPARATION PROCEDURE/SUMMARY DATA SHEET
SERIES #1, RUN #1

WHH
8/22/95

Fe Can with Lid #		Weights (g)								
		Digital Scale 1			Digital Scale 1		Digital Scale 2			
		Record Fe Can with Lid (g)			Record MgO (g)		Record CeO_2 (g)		Record ZrO_2 (g)	
		Can	Lid	Can + Lid	Target	Actual	Target	Actual	Target	Actual
284.52	11	73.44 +	7.64	= 81.08	201.43 ± 10	201.44	1 ± .05	1.01	1 ± .05	1.00
280.65	12	72.31 +	7.66	= 79.97	198.67 ± 10	198.67	1 ± .05	1.00	1 ± .05	1.00
284.89	13	73.60 +	7.59	= 81.19	201.70 ± 10	201.71	1 ± .05	1.01	1 ± .05	1.00
284.87	14	73.56 +	7.62	= 81.18	201.68 ± 10	201.69	1 ± .05	1.01	1 ± .05	1.00
286.85	15	74.07 +	7.68	= 81.75	203.09 ± 10	203.09	1 ± .05	1.00	1 ± .05	1.01
No. wt. measurements		15				3019.03 15		15.06 15		15.03 15

Packaging Requirements
Refer to ALDRICH quote attached

Health Safety Requirements
1. Follow MSDS handling instructions
2. Wear PPE per GA Tech practice. Consult the MSDS for general guidance

Special Requirements
1. Manually shake each closed can for 30 seconds after the ZrO_2/CeO_2 have been added
2. Record weight measurements to 4 significant figures
3. Mark tare weight of can with lid and "Series 1, Run 1" on each can
4. Store cans in air conditioned environment

NOTE: Nail punch three vent holes in the top of each can will be done in field as cans are fed

Major Equipment Requirements
(A) Ventilation hood
(B) 2-digital scales (one for ~1 lb measurements, one for ~1g measurements)

Date 9/11/95
Initial GJG

SURROGATE PREPARATION PROCEDURE/SUMMARY DATA SHEET
SERIES #1, RUN #1

WITH
8/22/95

Fe Can with Lid #		Weights (g)										
		Digital Scale 1			Digital Scale 1		Digital Scale 2				Digital Scale 2	
		Record Fe Can with Lid (g)			Record MgO (g)		Record CeO_2 (g)		Record ZrO_2 (g)		Record CaO (g)	
		Can	Lid	Can + Lid	Target	Actual	Target	Actual	Target	Actual	Target	Actual
234.10	1	72.96 +	7.57	= 80.53	95.85 ± 5	95.85	1 ± .05	1.00	1 ± .05	1.02	55.70 ± 5	55.70
236.39	2	73.63 +	7.48	= 81.11	96.54 ± 5	96.54	1 ± .05	1.03	1 ± .05	1.01	56.10 ± 5	56.71
234.68	3	73.25 +	7.49	= 80.74	96.10 ± 5	96.10	1 ± .05	1.01	1 ± .05	1.01	55.84 ± 5	55.84
235.85	4	73.44 +	7.71	= 81.15	96.58 ± 5	96.58	1 ± .05	1.04	1 ± .05	1.00	56.13 ± 5	56.13
234.28	5	73.03 +	7.55	= 80.58	95.91 ± 5	95.91	1 ± .05	1.03	1 ± .05	1.01	55.73 ± 5	55.74
235.53	6	73.56 +	7.47	= 81.03	96.44 ± 5	96.44	1 ± .05	1.01	1 ± .05	1.00	56.04 ± 5	56.05
234.92	7	73.05 +	7.42	= 80.47	95.78 ± 5	95.78	1 ± .05	1.01	1 ± .05	1.00	55.66 ± 5	56.67
235.43	8	73.54 +	7.45	= 80.99	96.39 ± 5	96.37	1 ± .05	1.01	1 ± .05	1.03	56.02 ± 5	56.02
236.12	9	73.69 +	7.54	= 81.23	96.68 ± 5	96.68	1 ± .05	1.00	1 ± .05	1.01	56.18 ± 5	56.20
232.23	10 (control sample)	72.28 +	7.59	= 79.87	95.06 ± 5	95.07	1 ± .05.	1.00	1 ± .05	1.01	55.24 ± 5	55.26
No. wt. measurements		10				961.32 10		10.11 10		10.10 10		560.32 10

Packaging Requirements

Refer to ALDRICH quote attached

Health Safety Requirements

1. Follow MSDS handling instructions
2. Wear PPE per GA Tech practice. Consult the MSDS for general guidance

Special Requirements

1. Manually shake each closed can for 30 seconds after the ZrO_2/CeO_2 have been added
2. Record weight measurements to 4 significant figures
3. Mark tare weight of can with lid and "Series 1, Run 2" on each can
4. Store cans in air conditioned environment

NOTE: Nail punch three vent holes in the top of each can will be done in field as cans are fed

Major Equipment Requirements

(A) Ventilation hood

(B) 2-digital scales (one for ~1 lb measurements, one for ~1g measurements)

Date 9/7/95

Initials GJG

SURROGATE PREPARATION PROCEDURE/SUMMARY DATA SHEET
SERIES #1, RUN #2

WITH
8/22/95

Fe Can with Lid #		Weights (g)												
		Digital Scale 1			Digital Scale 1		Digital Scale 2				Digital Scale 1			
		Record Fe Can with Lid (g)			Record MgO (g)		Record CeO, (g)		Record ZrO, (g)		Record CaO (g)		Record C (g)	
		Can	Lid	Can + Lid	Target	Actual	Target	Actual	Target	Actual	Target	Actual	Target	Actual
233.24	1	72.86 +	7.47	= 80.33	52.47 ± 5	52.47	1 ± .05	1.01	1 ± .05	1.01	55.48 ± 5	55.49	42.97 ± 5	42.99
236.16	2	73.82 +	7.56	= 81.38	53.16 ± 5	53.17	1 ± .05	1.00	1 ± .05	1.00	56.02 ± 5	56.02	43.56 ± 5	43.56
236.00	3	73.74 +	7.53	= 81.27	53.09 ± 5	53.09	1 ± .05	1.01	1 ± .05	1.00	56.13 ± 5	56.14	43.50 ± 5	43.50
230.08	4	73.95 +	7.35	= 81.30	53.11 ± 5	53.12	1 ± .05	1.02	1 ± .05	1.01	56.15 ± 5	56.14	43.52 ± 5	43.51
235.17	5	73.29 +	7.69	= 80.97	52.89 ± 5	52.90	1 ± .05	1.00	1 ± .05	1.00	55.92 ± 5	55.93	43.34 ± 5	43.34
231.27	6	71.98 +	7.67	= 79.65	52.03 ± 5	52.04	1 ± .05	1.03	1 ± .05	1.01	55.01 ± 5	55.01	42.63 ± 5	42.21
234.50	7	73.18 +	7.55	= 80.73	52.74 ± 5	52.75	1 ± .05	1.01	1 ± .05	1.01	55.76 ± 5	55.10	43.24 ± 5	43.24
235.05	8	73.45 +	7.47	= 80.92	52.86 ± 5	52.86	1 ± .05	1.06*	1 ± .05	1.03	55.89 ± 5	55.76	43.21 ± 5	43.30
236.38	9	73.76 +	7.65	= 81.41	53.18 ± 5	53.18	1 ± .05	1.00	1 ± .05	1.02	56.23 ± 5	56.26	43.57 ± 5	43.52
234.86	10	73.31 +	7.56	= 80.87	52.83 ± 5	52.83	1 ± .05	1.01	1 ± .05	1.00	55.85 ± 5	55.86	43.29 ± 5	43.29
237.80	11	74.24 +	7.64	= 81.88	53.49 ± 5	53.48	1 ± .05	1.01	1 ± .05	1.01	56.55 ± 5	56.59	43.83 ± 5	43.84
236.83	12	73.98 +	7.55	= 81.53	53.26 ± 5	53.25	1 ± .05	1.00	1 ± .05	1.09*	56.31 ± 5	56.32	43.64 ± 5	43.66
235.[illegible]	(control sample)	73.81 +	7.43	= 81.24	53.07 ± 5	53.07	1 ± .05	1.00	1 ± .05	1.01	56.11 ± 5	56.11	43.48 ± 5	43.49

Packaging Requirements
Refer to ALDRICH quote attached

Health Safety Requirements
1. Follow MSDS handling instructions
2. Wear PPE per GA Tech practice. Consult the MSDS for general guidance

Special Requirements
1. Manually shake each closed can for 30 seconds after the ZrO_2/CeO_2 have been added
2. Record weight measurements to 4 significant figures
3. Mark tare weight of can with lid and "Series 1, Run 3" on each can
4. Store cans in air conditioned environment

NOTE: Nail punch three vent holes in the top of each can will be done in field as cans are fed

Major Equipment Requirements
(A) Ventilation hood
(B) 2-digital scales (one for ~1 lb measurements, one for ~1g measurements)

Date 9/8/95
Initials GJG

SURROGATE PREPARATION PROCEDURE/SUMMARY DATA SHEET
SERIES #1, RUN #3

WHH
8/22/95

SURROGATE PREPARATION PROCEDURE/ SUMMARY DATA SHEET

SERIES #2

Factors----------->				*ZrO2= 0.9303797*		*CeO2= 0*		*CaO= 0.6962025*		*C= 0.5379747*	
Can Num	Fe(g) Can	Fe(g) Lid	Fe(g) Can + Lid	ZrO2(g) Target	ZrO2(g) Actual	CeO2(g) Target	CeO2(g) Actual	CaO(g) Target	CaO(g) Actual	C Target	C Actual
Percent Composition-->			*31.60*	*29.40*		*0%*		*22.00*		*17.00*	
1	72.67	7.67	80.34	74.75	74.76	1.00	1.00	55.93	55.94	43.22	43.23
2	73.57	7.46	81.03	75.39	75.38	1.00	1.01	56.41	56.41	43.59	43.60
3	73.51	7.64	81.15	75.50	75.49	1.00	1.01	56.50	56.51	43.66	43.65
4	73.48	7.51	80.99	75.35	76.36	1.00	1.00	56.39	56.39	43.57	43.57
5	73.63	7.58	81.21	75.56	75.55	1.00	1.00	56.54	56.55	43.69	43.68
6	73.82	7.71	81.53	75.85	75.85	1.00	1.00	56.76	56.76	43.86	43.87
7	73.70	7.66	81.36	75.70	75.70	1.00	1.00	56.64	56.66	43.77	43.78
8	74.02	7.51	81.53	75.85	75.85	1.00	1.02	56.76	56.76	43.86	43.88
9	74.03	7.47	81.50	75.83	75.82	1.00	1.01	56.74	56.75	43.84	43.86
10	73.80	7.49	81.29	75.63	75.63	1.00	1.00	56.59	56.60	43.73	43.74
11	73.42	7.56	80.98	75.34	75.34	1.00	1.02	56.38	56.41	43.57	43.57
12	73.75	7.74	81.49	75.82	75.81	1.00	1.01	56.73	56.74	43.84	43.84
13	72.62	7.46	80.08	74.50	74.49	1.00	1.00	55.75	55.76	43.08	43.08
14	73.74	7.39	81.13	75.48	75.47	1.00	1.02	56.48	56.50	43.65	43.64
15	72.96	7.63	80.59	74.98	74.98	1.00	1.00	56.11	56.13	43.36	43.38
16	73.75	7.55	81.30	75.64	75.65	1.00	1.08	56.60	55.65	43.74	43.74
17	74.06	7.46	81.52	75.84	75.86	1.00	1.01	56.75	56.77	43.86	43.86
18	73.57	7.53	81.10	75.45	75.45	1.00	1.02	56.46	56.46	43.63	43.65
19	74.28	7.54	81.82	76.12	76.13	1.00	1.01	56.96	56.98	44.02	44.02
20	73.78	7.72	81.50	75.83	75.81	1.00	1.00	56.74	56.75	43.84	43.86
21	73.04	7.56	80.60	74.99	74.99	1.00	1.00	56.11	56.15	43.36	43.36
22	72.02	7.60	79.62	74.08	74.08	1.00	1.00	55.43	55.42	42.83	42.85
23	73.37	7.47	80.84	75.21	75.21	1.00	1.00	56.28	56.27	43.49	43.52
24	73.14	7.67	80.81	75.18	75.18	1.00	1.02	56.26	56.28	43.47	43.48
25	73.44	7.47	80.91	75.28	75.27	1.00	1.02	56.33	56.33	43.53	43.51
26	73.41	7.64	81.05	75.41	75.41	1.00	1.02	56.43	56.42	43.60	43.61
27	72.45	7.46	79.91	74.35	74.35	1.00	1.00	55.63	55.63	42.99	43.00
28	73.52	7.54	81.06	75.42	37.96	1.00	1.00	56.43	56.46	43.61	43.63
TOTAL FED	1983.88	204.02	2187.90	2035.58	1999.07	27.00	27.28	1523.22	1522.50	1177.03	1177.23
TOTAL DONE	2056.55	211.69	2268.24	2110.32	2073.83	28.00	28.28	1579.15	1578.44	1220.26	1220.46

NOTE: Can #1, Given to Luther Gibson for safekeeping, 19 Sep 95

Chemicals mixed by: GEOVING J. GERARD Date: 18 Sep, 95 Initials: GJG

C

APPENDIX D

GEORGIA TECH SURROGATE TESTING PROCESS DATA FIELD DATA SHEETS AND PROCESS DATA SUMMARY

GEORGIA TECH SURROGATE TESTING
PROCESS DATA FIELD DATA SHEETS
AND PROCESS DATA SUMMARY

Test Phase	Date
Hot Shakedown	9/11/95/ (6 pages)
Pre-Heat	9/12/95 (6 pages)
Series 1, Run 1, Surrogate	9/12/95 (4 pages)
Series 1, Run 2 Surrogate	9/12/95 (2 pages)
Series 1, Run 3 Surrogate	9/12/95 (3 pages)
Cold Shakedown	9/18/95 (2 pages)
Pre-Heat	9/19/95 (5 pages)
Series 2 Surrogate	9/19/95 (6 pages)

Event Description/Time	Start Torch 1450	1457	1502	1505	1508	1511	1516
Torch Time ON/OFF	Torch 'ON' 1450						
Pyrometer Temp. °C							
Torch Position, in	12	12	12	12	12	12	12
Torch Voltage, V/ Amps, A	480/138	400/170	440/180	460/180	400/200	400/200	400/200
Torch Carrier Gas Pressure (PSIG)	40-50	34	38	45	50	58	50
Center Thermocouple, °C	26	280	313	359	503	675	806
Furnace Pressure, in W.C.	-0.4	-0.45	-0.4	-0.45			-0.45
Notes: I.D. Fan Inlet Valving 100% Open Scrubber Recycle Pump Head = 36 psig/vacuum 0 in Hg		White bubble appeared w/orange hot surface	Molten white w/orange background		Constant air pressure adjustment (manual) required	Reactor skin temp delayed gas temp and then started to ramp up	Torch center forms white molten regime
Scrubber pH							
Run time (min)							

Initial: WHH
Date: 9/11/95

TEST PHASE: HOT SHAKEDOWN

Event Description/Time	1519	Shutdown Torch 1525	1536	Moved Torch Lower 1532	1534	1536	Moved Torch Higher 1536.30
Torch Time ON/OFF		Torch 'ON' 1526.30					
Pyrometer Temp. °C		880					
Torch Position, in	12	12	12	9	9	9	10.5
Torch Voltage, V/ Amps, A	400/200		380/200		400/200	360/200	380/200
Torch Carrier Gas Pressure (PSIG)	58		55		45	38	
Center Thermocouple, °C	857	944 (range)	1025		1800	>3241	
Furnace Pressure, in W.C.	-0.4		-.4 to -.45	-.4 to -.45	-0.45		
Notes		MUSA took pyrometer reading w/oversight from Bob Newsom		Melt hole visible from camera– direct impinge-ment on crucible		Witnessed excessive Tc increase	
Scrubber pH							
Run time (min)							

Initial WHH
Date: 9/11/95

TEST PHASE: HOT SHAKEDOWN

Event Description/Time	1542	Torch Shutdown 1544	1551	1557	Torch Shutdown 1605	1615	1620
Torch Time ON/OFF		Torch 'ON' 1545.30			Torch 'ON' 1605.45		
Pyrometer Temp. °C		1035			1211		1400
Torch Position, in	10.5	10.5	10.5	10.5	10.5	10.5	10.5
Torch Voltage, V/ Amps, A	380/200		420/200		400/200		
Torch Carrier Gas Pressure (PSIG)	42		40	48	58		
Center Thermocouple, °C	>3180		Checked Channel 3 >2500	Channel 3 >4930			
Furnace Pressure, in W.C.			-0.45	-0.4		-0.45	
Notes	High scatter in Tc readings/ spalling in crucible noticed/ could be coating	On torch shutdown, pyrometer temp dropped very fast	Requested scrubber pH be checked	Requested 50% NaOH (caustic) be added			
Scrubber pH				2.5	Added 2-250ml 2-400ml	5.6	
Run time (min)							

Initial: WHH
Date: 9/11/95

TEST PHASE: HOT SHAKEDOWN

Event Description/Time	1625	1631	Shut Torch Down 1631.30	1647	1651	Add Fe Can 1 1655	1700
Torch Time ON/OFF			Torch 'ON' 1632.30				
Pyrometer Temp. °C	1322	1349	1122		1242		1460
Torch Position, in	10.5	10.5	10.5	10.5	10.5	10.5	10.5
Torch Voltage, V/ Amps, A							
Torch Carrier Gas Pressure (PSIG)							
Center Thermocouple, °C							
Furnace Pressure, in W.C.			-0.4				
Notes			Noticed smoke from reactor-gaskets burning exposed to air covered by insulation	Per video, losing iron coating on crucible-decide to add Fe	Made up cans w/nuts weighing 5,554.5g or 5.5 Kg	Fe added in form of can w/nuts	Nuts molten after a couple minutes
Scrubber pH	10	Added 2-400 ml caustic		7.3			
Run time (min)					118		

Initial: WHH
Date: 9/11/95

TEST PHASE: HOT SHAKEDOWN

Event Description/Time	Added Fe 1703	1709	Shut Torch Down 1710.30		1720	1722	1725
Torch Time ON/OFF			Torch 'ON' 1711.15				
Pyrometer Temp. °C		1392	1310			1417	1411
Torch Position, in	10.5	10.5	10.5	10.5	10.5	10.5	10.5
Torch Voltage, V/ Amps, A			380/200				
Torch Carrier Gas Pressure (PSIG)			58				
Center Thermocouple, °C							
Furnace Pressure, in W.C.			-0.4				
Notes	Added second can of nuts (total can 1 & can 2 weight ~5554.5g)		Requested 250 ml NaOH addition				
Scrubber pH			7.3	Added 250ml NaOH	7		
Run time (min)						147	

Initial: WHH
Date: 9/11/95

TEST PHASE: HOT SHAKEDOWN

Event Description/Time	Torch Shutdown 1725.30						
Torch Time ON/OFF							
Pyrometer Temp. oC	1347						
Torch Position, in	10.5						
Torch Voltage, V/ Amps, A							
Torch Carrier Gas Pressure (PSIG)							
Center Thermocouple, oC							
Furnace Pressure, in W.C.							
Notes	In summary, the system operated @~80KW for 150 minutes	Total 50 wt% NaOH fed 9/11/95 2-250ml 5-400ml	Based on pyrometer data, steady state temp setpoint ~1400°C				
Scrubber pH							
Run time (min)							

Initial: WHH
Date 9/11/95

TEST PHASE: HOT SHAKEDOWN

Event Description/Time	915	950	1004	Start Torch 1010 45	1017	1029	Lower Torch 1030 40
Torch Time ON/OFF							
Pyrometer Temp. °C							
Torch Position, in	10.5	10.5	10.5	10.5	10.5	10.5	9
Torch Voltage, V/ Amps, A				380/150	380/200	380/200	
Torch Carrier Gas Pressure (PSIG)				35-52			
Center Thermocouple, °C						480	
Furnace Pressure, in W.C.		-0.75			-0.5	-0.7	-0.7
Notes:	Took scrubber water sample for baseline after running >20 min. Added rain cap to inlet valve to I.D. fan		Set-up automatic torch air pressure control	Tried to run at ~700KW for a few minutes	Observed small melt surface		
Scrubber pH		7.9					
Run time (min)					6.5		20

Initial: WHH
Date: 9/12/95

TEST PHASE: PRE-HEAT

Event Description/Time	1045	1054	1056	Raised Torch 1100	1110	1114	1116
Torch Time ON/OFF							
Pyrometer Temp. °C							
Torch Position, in	9	9	9	10.5	10.5	10.5	10.5
Torch Voltage, V/ Amps, A	370/200	380/200			380/200		
Torch Carrier Gas Pressure (PSIG)	34-54				36-54		
Center Thermocouple, °C	1050	>2800			>5600		
Furnace Pressure, in W.C.	-0.7	-0.7		-.6	-0.5		
Notes		Not a large melt surface obvious	Added 250ml caustic (NaOH)	Lou Circeo arrived @ site	Took off rain cap from inlet valve– slight higher pressure in primary experienced		Added 250 ml NaOH and stirred
Scrubber pH	7.2			7		6.2	
Run time (min)	36	~42		50.5			

Initial: WHH
Date: 9/12/95

TEST PHASE: PRE-HEAT

Event Description/Time	1121	1129	Lowered Torch 1131		Raised Torch 1134	1136	1140
Torch Time ON/OFF							
Pyrometer Temp. °C							
Torch Position, in	10.5	10.5	7	7	9	9	9
Torch Voltage, V/ Amps, A	380/200	380/200	380/200				380/200
Torch Carrier Gas Pressure (PSIG)	37-54	37-54	37-54				37-54
Center Thermocouple, °C	>6000	>6260					
Furnace Pressure, in W.C.	-0.5	-0.6					-0.6
Notes			With video, observed raw graphite surface with immediate need to raise torch	Did also see melting w/high blow-out or fluidity	Saw graphite surface slowly be recovered with melt	With stick wedge, pushed torch to one side to melt nut added 9/11/95 that was not melted	
Scrubber pH		1133 +250ml 6.2					
Run time (min)		78	80		83.5	85	90

Initial: WHH
Date: 9/12/95

TEST PHASE: PRE-HEAT

Event Description/Time	Lowered Torch 1144	1146	Raised Torch 1147	Raised Torch 1148	Added Can 1 1149	Added Can 2 1150	Added Can 3 1150.3
Torch Time ON/OFF							
Pyrometer Temp. °C							
Torch Position, in	7	7	9	10.5	10.5	10.5	10.5
Torch Voltage, V/ Amps, A							
Torch Carrier Gas Pressure (PSIG)							
Center Thermocouple, °C							
Furnace Pressure, in W.C.							
Notes	Lowered to torch to ensure nut completely melted	Observed with camera that nut had fallen into melt			To supplement Fe seed bath 13 empty, crushed cans/ without lid/were added– starting w/can 1		
Scrubber pH	1142 +250ml 6.6						
Run time (min)	93			97			99.5

Initial: WHH
Date: 9/12/95

TEST PHASE: PRE-HEAT

Event Description/Time	1152.3	1155	Add Can 4 1158 40	Add Can 5 1159.45	Added Cans 6 & 7 1201.15	Added Cans 8 & 9 1201.50	Added Cans 10 & 11 1202.09
Torch Time ON/OFF							
Pyrometer Temp. °C		1415					
Torch Position, in	10.5	10.5	10.5	10.5	10.5	10.5	10.5
Torch Voltage, V/ Amps, A	380/200		380/200				
Torch Carrier Gas Pressure (PSIG)	30-42						
Center Thermocouple, °C							
Furnace Pressure, in W.C.							
Notes	Changed to lower automatic air pressure range—no change in KW		Can weight w/o lid is 72.9g		Geoving Gerard started to take pH/scrubber water pressure data		
Scrubber pH			1157 +250ml 7.4				
Run time (min)			108		110		

Initial: WHH
Date: 9/12/95

TEST PHASE: PRE-HEAT

Event Description/Time	Added Cans 12 & 13 1203	1208					
Torch Time ON/OFF							
Pyrometer Temp. oC		1500					
Torch Position, in	10.5	10.5					
Torch Voltage, V/ Amps, A	375/200	360/200					
Torch Carrier Gas Pressure (PSIG)							
Center Thermocouple, oC							
Furnace Pressure, in W.C.	-0.65						
Notes	Could see uniform melt	Bath looked 'good' for feeding surrogate; exhanged out video cassette tape					
Scrubber pH							
Run time (min)	112	118					

Initial: WHH
Date: 9/12/95

TEST PHASE: PRE-HEAT

Event Description/Time	Surrogate Can 1 fed 1212	Can 2 fed 1218 30	Can 3 fed 1224 20	1225 45	Can 4 fed 1230 40	Can 5 fed 1234 06	1235 40
Torch Time ON/OFF							
Pyrometer Temp. °C		1535		1603			1621
Torch Position, in	10.5	10.5	10.5	10.5	10.5	10.5	10.5
Torch Voltage, V/ Amps, A						360/200	
Torch Carrier Gas Pressure (PSIG)				28-40	28-40		
Center Thermocouple, °C							
Furnace Pressure, in W.C.							-0.6
Notes:	Started surrogate testing–with can 1 feed, powder appeared to cloud video for a few seconds	Appeared to see some MgO mixing with Fe, viscosity increasing/ less fluid					
Scrubber pH	1212 +250ml 7.3			1227 +250ml 7.4			
Run time (min)	122	127.5	133.5	134.3	139.6	143.2	

Initial: WHH
Date: 9/12/95

TEST PHASE: SERIES 1, RUN 1

Event Description/Time	Can 6 fed 1239.40	Can 7 fed 1242.50	1245.50	Can 8 fed 1246.30	Can 9 fed 1250.25	Can 10 fed 1253.27	1254
Torch Time ON/OFF							
Pyrometer Temp. °C				1563			1570
Torch Position, in	10.5	10.5	10.5	10.5	10.5	10.5	10.5
Torch Voltage, V/ Amps, A		380/200	360/200			370/200	400/200
Torch Carrier Gas Pressure (PSIG)	34-48	33-49		34-49			
Center Thermocouple, °C							
Furnace Pressure, in W.C.							
Notes	Increased air pressure range						
Scrubber pH		1242 +250ml 7.4					
Run time (min)	149.2	152.1			159.7	163	

Initial: WHH
Date: 9/12/95

TEST PHASE: SERIES 1, RUN 1

Event Description/Time	Can 11 fed 1259	1304.3	Can 12 fed 1306.28	Can 13 fed 1313.02	1314.3	Can 14 fed 1318.30	Lowered torch 1324.30
Torch Time ON/OFF							
Pyrometer Temp. °C		1652			1690		1609
Torch Position, in	10.5	10.5	10.50	10.5	10.5	10.5	9
Torch Voltage, V/ Amps, A	400/200				400/200		
Torch Carrier Gas Pressure (PSIG)							
Center Thermocouple, °C							
Furnace Pressure, in W.C.					-0.6		
Notes					Furnace skin was observed to be red hot	Furnace was wrapped with insulation for this test (very extensively) skin cherry red	The torch was lowered to try to form a melt
Scrubber pH	1257 +250ml 7.4			1312 +250ml 7.4			
Run time (min)	168.1		175.60	182.3		187.8	194

Initial: WHH
Date: 9/12/95

TEST PHASE: SERIES 1, RUN 1

Event Description/Time	Lowered Torch (1331.50)	Lowered Torch 1333.20	Lowered Torch 1338.30	Lowered Torch 1343.15	1345.50	Raised Torch 1349.30	
Torch Time ON/OFF							
Pyrometer Temp. °C	1632				1584.00		
Torch Position, in	7	6	5	4	4.00	10.5	
Torch Voltage, V/ Amps, A		400/200					
Torch Carrier Gas Pressure (PSIG)		35-52					
Center Thermocouple, °C							
Furnace Pressure, in W.C.	-0.6						
Notes			Lowered torch to 5 in, still trying to ascertain melt condition in furnace crucible w/video camera		Noted the torch cooling water temperature was close to shut-off setpoint of 91°F (started at 82°F)	Splatter at low torch position caused crud build-up on torch end; raising the torch did lower water temperature to 87°F	
Scrubber pH	1330 +500ml 7.0			1342 +250ml 7.4			
Run time (min)	201.2	202	-207	212.5		219	

Initial: WHH
Date: 9/12/95

TEST PHASE: SERIES 1, RUN 1

Event Description/Time	Can 1 fed 1355.12	Can 2 fed 1400.40	Can 3 fed 1406.40	Can 4 fed 1412.25	1414.3	Can 5 fed 1417.40	Can 6 fed 1420.38
Torch Time ON/OFF							
Pyrometer Temp. °C					1607		
Torch Position, in	10.5	10.5	10.5	10.5	10.5	10.5	10.5
Torch Voltage, V/ Amps, A	400/200		400/200	400/200			400/200
Torch Carrier Gas Pressure (PSIG)							34-49
Center Thermocouple, °C							
Furnace Pressure, in W.C.		-0.55					-0.65
Notes:	Noted white halo around the edges, start Series 1, Run 2		Noted white halo increasing in diameter	Yellow/white area increasing			Yellow/white area on periphery
Scrubber pH	1357 +250ml 7.4			1412 +250ml 7.3			
Run time (min)	224.3	229.8	236	241.5		246.8	249.8

Initial: WHH
Date: 9/12/95

TEST PHASE: SERIES 1, RUN 2

Event Description/Time	1426	Can 7 fed 1428.07	Can 8 fed 1432.20	1434	Can 9 fed 1435	1442	
Torch Time ON/OFF							
Pyrometer Temp. °C	1591			1605			
Torch Position, in	10.5	10.5	10.5	10.5	10.5	10.5	
Torch Voltage, V/ Amps, A			390/200		390/200		
Torch Carrier Gas Pressure (PSIG)					34-49		
Center Thermocouple, °C							
Furnace Pressure, in W.C.							
Notes	Noticed fluid/lower viscosity motion, clearly molten on top, right section of screen				Fluid throughout entire top surface		
Scrubber pH	1427 +250ml 7.3					1445 +500ml 7.1	
Run time (min)		257.3	261.6		264.2		

Initial: WHH
Date 9/12/95

TEST PHASE: SERIES 1, RUN 2

Event Description/Time	Raised Torch 1446.24	Can 1 fed 1446.50	Can 2 fed 1449.08	Can 3 fed 1452	Can 4 fed 1454.45	Can 5 fed 1456.38	Can 6 fed 1500.36
Torch Time ON/OFF							
Pyrometer Temp. °C							
Torch Position, in	11.5	11.5	11.5	11.5	11.5	11.5	11.5
Torch Voltage, V/ Amps, A	400/200				400/200		
Torch Carrier Gas Pressure (PSIG)	34-50				34-50		
Center Thermocouple, °C							
Furnace Pressure, in W.C.							
Notes:		Initial or first time carbon is fed with MgO/CaO surrogate, start Series 1, Run 3		Melt area more obvious, white dust cloud appeared for 6-7 seconds upon feeding		Dust cloud ~6-7 seconds periodically viewed melt from top of vessel using welding helmet UV prot. glass	Moving camera to view directly shows up as interruption on VCR film
Scrubber pH						1457 +250ml 7.5	
Run time (min)		275.7	278.3	281	283.9	286	289.7

Initial: WHH
Date: 9/12/95

TEST PHASE: SERIES 1, RUN 3

Event Description/Time	1502.45	Can 7 fed 1503.20	Can 8 fed 1505.26	Can 9 fed 1507.40	1509	Can 10 fed 1510.25	Can 11 fed 1512.20
Torch Time ON/OFF							
Pyrometer Temp. °C	1700				1658		
Torch Position, in	11.5	11.5	11.5	11.5	11.5	11.5	11.5
Torch Voltage, V/ Amps, A			400/200				
Torch Carrier Gas Pressure (PSIG)			36-50				
Center Thermocouple, °C							
Furnace Pressure, in W.C.			-.55				
Notes					Noted the 'Pizza' look on the video is due to solid build-up on glass, actually entire surface is white hot, melt formed		
Scrubber pH							1512 +250ml 7.4
Run time (min)		292.4	294.5	296.8		299.5	301.4

Initial: WHH
Date: 9/12/95

TEST PHASE: SERIES 1, RUN 3

Event Description/Time	Can 12 fed 1513.13	Shutdown Torch 1518	Post Test Note		Post Test Note	
Torch Time ON/OFF		Torch 'OFF'				
Pyrometer Temp. °C						
Torch Position, in	11.5	11.5				
Torch Voltage, V/ Amps, A						
Torch Carrier Gas Pressure (PSIG)						
Center Thermocouple, °C						
Furnace Pressure, in W.C.						
Notes	Target running 5 min at steady-state		Checked the scrubber water sump with dip tube to check build-up and found little material, pressure head stayed steady throughout the test at 36 psig, plan to replace sock filter for Series 2 test		Pulled torch immediately after test and visually inspected/photographed from standpoint of decontamination/ solids build-up	
Scrubber pH			1522 7.4	1543 7.1		
Run time (min)	302.3	308.2				

Initial: WHH
Date: 9/12/95

TEST PHASE: SERIES 1, RUN 3

Event Description/Time	Pre-Test Note	Pre-Test Note	Pre-Test Note
Torch Time ON/OFF			
Pyrometer Temp. °C			
Torch Position, in			
Torch Voltage, V/ Amps, A			
Torch Carrier Gas Pressure (PSIG)			
Center Thermocouple, °C			
Furnace Pressure, in W.C.			
Notes:	Added 4538.4g Fe to crucible, 3 cans filled with washers, 1 can larger washers, 2 cans small washers. Washers had oil film on them, did not clean	Noted the ZrO_2 total shipment was 2 Kg + 100g, structured Series 2 based on ZrO_2 available	Per guidance from Bob and Lamar (GA Tech), we calibrated the torch power/flame throw length by starting torch outside primary vessel (in ambient air) and visually inspected, checked electrode wear as a function of air pressure.
Notes: (Cont'd)			Optimum setting equivalent to 25 to 35 psig/several inches flame length
Scrubber pH			
n time (min)			

Initial: WHH
Date: 9/18/95

TEST PHASE: COLD SHAKEDOWN

Event Description/Time	Pre-Test Note	Pre-Test Note
Torch Time ON/OFF		
Pyrometer Temp. °C		
Torch Position, in		
Torch Voltage, V/ Amps, A		
Torch Carrier Gas Pressure (PSIG)		
Center Thermocouple, °C		
Furnace Pressure, in W.C.		
Notes	Ran vacuum system and water recycle system 8:30-9:00 with fan inlet valving 100% open, vacuum = -.5 in W.C., closed valving at fan inlet and ran vacuum at -2 in W.C. to try to remove loose settled solids from piping and drive to recycle tank.	Completed pitot tube traverse and result was 0.005 in W.C. velocity head which was the same as initial cold flow check (~80 SCFM), check done at riser upstream of I.D. fan
Notes (Cont'd)	Ran from 900-1000 and then changed filter (sock type) in recycle loop	
Scrubber pH		
Run time (min)		

Initial: WHH
Date: 9/19/95

TEST PHASE: COLD SHAKEDOWN

Event Description/Time	Start Torch 1001.07	1006	1012 32	1017	1022.3	1028 15	1031 35
Torch Time ON/OFF	Torch 'ON'						
Pyrometer Temp. °C							
Torch Position, in	9	9	9	9	9	9	9
Torch Voltage, V/ Amps, A	400/170	400/170	420/185	425/185	420/185	425/185	425/185
Torch Carrier Gas Pressure (PSIG)	25-35	25-35	27-36		27-36	27-36	27-36
Center Thermocouple, °C							
Furnace Pressure, in W.C.	-0.6	-0.6		-0.6	-0.6		
Notes:	Could view the washers melting in immediate area where plasma envelope existing, temp. profile extreme	Hooked up automatic (A) pH meter, will take manual (M) as well	Tried to reposition torch over unmelted cans, manually used push rod	Melt forming over entire crucible bottom surface area	Can see fluid melt (vibration effect) on outer edges (turbulent) partial can wall still visible, Fe depth increasing	No base crucible surface area visible, melt formation with skeletal can wall still visible	Can fell into melt
Scrubber pH					1023 M - A 8.1 - 8.91		
Run time (min)		6	11.4	15.9	21.4	27.1	30.4

Initial: WHH
Date: 9/19/95

TEST PHASE: PRE-HEAT

Event Description/Time	1034	Fe Can 1 Addition 1037.22	Fe Can 2 Addition 1039.15	Fe Can 3 Addition 1041	1047	Fe Can 4 Addition 1047.50	Fe Can 5 Addition 1050
Torch Time ON/OFF							
Pyrometer Temp. °C					1550		
Torch Position, in	9	9	9	9	9	9	9
Torch Voltage, V/ Amps, A		440/185		415/200		420/200	
Torch Carrier Gas Pressure (PSIG)						27-35	
Center Thermocouple, °C							
Furnace Pressure, in W.C.							-0.6
Notes	Tried to feed Fe Can No. 1, had blockage in plunger/ valve area	Fe Can 1 fed, 1470g, had to open side hatch and use probe to push	Fe Can 2 fed, 1457.2g	Fe Can 3 fed, 1542.1g		Fe Can 4 fed, 1613.8g	Fe Can 5 fed, 1631.6g, could see shadow of unmelted can
Scrubber pH				1043 7.8 - 8.63 M - A			
Run time (min)		36.4	38.3	40		47	49.2

Initial: WHH
Date: 9/19/95

TEST PHASE: PRE-HEAT

Event Description/Time	1054 30	Fe Can 6 Addition 1059	Fe Can 7 Addition 1101	1102.30	1108.20	Raise Torch 1109.03	1116 40
Torch Time ON/OFF							
Pyrometer Temp. °C	1520.00			1422.00			1500.00
Torch Position, in	9	9	9	9	9	11	11
Torch Voltage, V/ Amps, A		400/200	420/200		420/200	420/200	420/200
Torch Carrier Gas Pressure (PSIG)				27-36	27-36	27-36	27-36
Center Thermocouple, °C	900.00			946.00	964.00		
Furnace Pressure, in W.C.						-0.6	
Notes	Can see splatter on glass lens so Fe deposition is occurring relatively quick into this run	Fe Can 6 fed, 1644.2g	Fe Can 7 fed, 1630g	Tried putting UV filter on view point, molten pool 4" diameter, outer periphery meter already crusted, can see 2 cans not melted, (Cans 6,7)			
Scrubber pH		1058 M - A 7.7 - 8.48				1113 7.6 - 8.31 M - A	
Run time (min)		58.1	60		67.6	68.1	76

Initial: WHH
Date 9/19/95

TEST PHASE: PRE-HEAT

Event Description/Time	Fe Can 8 Addition 1118.14	Fe Can 9 Addition 1118 47	1122	Lowered Torch 1126.50	1128	1130	Note
Torch Time ON/OFF							
Pyrometer Temp. °C			1410		1530	1560	
Torch Position, in	11	11	11	9	9	9	9
Torch Voltage, V/ Amps, A				420/200			
Torch Carrier Gas Pressure (PSIG)					27-39		26-39
Center Thermocouple, °C		927	870		860	866	
Furnace Pressure, in W.C.					-0.6		
Notes	Fe Can 8 fed, 1550.1g	Fe Can 9 fed, 1618.6g	Noted drop in temperature readings @ 11" position, also, suspect plunger/ valve interfered with can addition @ 9"				Channel 5 Tc appears to work as does separate unit set-up. stopped Fe addition @ 3 cans + 9 cans (reheat) @ 41.22 lbs or 2" depth
Scrubber pH					1128 +250ml 7.4 - 8.18 M - A		
Run time (min)	77.1	77.8		86		89	

Initial: WHH
Date: 9/19/95

TEST PHASE: PRE-HEAT

Event Description/Time	1138	1141 50	1148	1150.30			
Torch Time ON/OFF							
Pyrometer Temp. °C	1560	1560		1500			
Torch Position, in	9	9					
Torch Voltage, V/ Amps, A		420/200					
Torch Carrier Gas Pressure (PSIG)							
Center Thermocouple, °C	972	1004		1030			
Furnace Pressure, in W.C.							
Notes			Changed glass lens on reactor for camera visibility to have new for Series 2 testing				
Scrubber pH		1143 8 - 8.8 M - A					
Run time (min)		100.7					

Initial: WHH
Date: 9/19/95

TEST PHASE: PRE-HEAT

Event Description/Time	Surrogate Can 2 Fed 1156.30	Surrogate Can 3 Fed 1202	Raise Torch 1203	1205.20	Surrogate Can 4 Fed 1207.40	1211	Surrogate Can 5 Fed 1213.20
Torch Time ON/OFF							
Pyrometer Temp. °C	1450		1450			1400	
Torch Position, in	9	9	11	11	11	11	11
Torch Voltage, V/ Amps, A	400/200						
Torch Carrier Gas Pressure (PSIG)	26-39						
Center Thermocouple, °C	1019			1021		1019	1023
Furnace Pressure, in W.C.	-						
Notes:	Surrogate Can 1 was retained as sample archived; color changed somewhat from previous test	Color change	Raised torch to eliminate plunger rod interference with torch; temp. drops with surrogate can feed		Shook the can prior to addition; could see powder dispersion per video, mixing not done prior to this time		Noted the camera lens should be water cooled with jacket
Scrubber pH	1158 7.6 - 8.3 M - A						1213 +250ml 7.1 - 7.91 M - A
Run time (min)	115.5	121.2	122	124.3	126.7	128.9	132.5

Initial: WHH
Date: 9/19/95

TEST PHASE: SERIES 2

Event Description/Time	1216.20	Surrogate Can 6 Fed 1218.40	Surrogate Can 7 Fed 1223	1225	Surrogate Can 8 Fed 1228	1230.50	Surrogate Can 9 Fed 1233.35
Torch Time ON/OFF							
Pyrometer Temp. °C	1400			1500	1500		
Torch Position, in	11	11	11	11	11	11	11
Torch Voltage, V/ Amps, A		400/200	420/200				
Torch Carrier Gas Pressure (PSIG)			25-40				
Center Thermocouple, °C		1027	1028			1036	1041
Furnace Pressure, in W.C.							
Notes		Cans definitely feed in smoother @ 11" versus 9" position	Saw distinctly different color, more orange with this mix		Noted positive pressure flux for a couple seconds with can feed, could hear cracking sound		After 90 second post can feed, saw positive pressure and heard view glass click
Scrubber pH					1228 +250 ml 7.3 - 7.95 M - A		
Run time (min)		137.6	142	144.1	147		152.5

Initial: WHH
Date: 9/19/95

TEST PHASE: SERIES 2

Event Description/Time	Surrogate Can 10 Fed 1239.30	Surrogate Can 11 Fed 1246	1249.15	Surrogate Can 12 Fed 1252.30	1254.2	Surrogate Can 13 Fed 1259.20	Surrogate Can 14 Fed 1304
Torch Time ON/OFF							
Pyrometer Temp. °C			1520		1580	1600	
Torch Position, in	11	11	11	11	11	11	11
Torch Voltage, V/ Amps, A		420/200	420/200				
Torch Carrier Gas Pressure (PSIG),			25-40				
Center Thermocouple, °C		1060	1060		1056	1057	1064
Furnace Pressure, in W.C.							
Notes	Camera lens falls up/down after 70 to 90 seconds post can feed, heard click then 20 second later pressure flux	Determined gas in can is expanding, will punch nail vent holes in cans	Camera lens fouled significantly			Venting can with holes has not completely eliminated pressure pulse, increase XS area of hole	
Scrubber pH						1258 +250 ml 7.1 - 7.87 M - A	
Run time (min)	158.4	165.1		171.7	173.4	178.5	183

Initial: WHH
Date: 9/19/95

TEST PHASE: SERIES 2

Event Description/Time	1308	Surrogate Can 15 Fed 1310	1313.20	Surrogate Can 16 Fed 1315.05	Surrogate Can 17 Fed 1322.35	Surrogate Can 18 Fed 1328	Surrogate Can 19 Fed 1334.30
Torch Time ON/OFF							
Pyrometer Temp. °C	1620						
Torch Position, in	11	11	11	11	11	11	11
Torch Voltage, V/ Amps, A			420/200				
Torch Carrier Gas Pressure (PSIG)							
Center Thermocouple, °C		1065	1074	1076	1079	1090	1125
Furnace Pressure, in W.C.							
Notes				Plunger has been sticking routinely, suspect blockage due to Fe splatter	Did not feed immediately, stuck for 90 seconds, plugging in Method 5 filter occurred, shutdown Method 5 @ 1329 per trailer clock,	With valve open, there is dusting, could be rx occurring	Much 'flashing' occurring on video underneath dirty lens, GA Tech wants to stop at 20 cans, I said 'OK', severe plunger
Scrubber pH			1313 +250 ml M - A 7.1 - 7.81			1328 +250 ml M - A 7.1 - 7.79	
Run time (min)		188		194	201.6	205.9	213.5

Initial: WHH
Date: 9/19/95

TEST PHASE: SERIES 2

Event Description/Time	1337.05	Surrogate Can 20 Fed 1339.40	Note	Note	1348	1354	1357.20
Torch Time ON/OFF							
Pyrometer Temp. °C	1650				1690.00	1670	
Torch Position, in	11	11					
Torch Voltage, V/ Amps, A							
Torch Carrier Gas Pressure (PSIG)							
Center Thermocouple, °C		1128					
Furnace Pressure, in W.C.							
Notes		Stopped surrogate feed based on possibility of plunger sticking and pressure pulsation/ vibration of melt	Glass popping partially due to single versus double lens, no manometer change after venting cans	Last can did not feed, melting occurring @ plunger/crue interface	Residuals in plunger feeder tube will be scraped and placed in crucible	Scrubber header downstream of spool piece will be flushed to recycle tank prior to sampling for release	
Scrubber pH				1343 +250 ml M - A 7.1 - 7.81			1358 +250 ml 7.1 - 7.83 M - A
Run time (min)	216.2	218.6					236.2

Event Description/Time	1400.23	Shut Torch Down 1401.29	1402.20	1404.30	1406.30	1408.40	
Torch Time ON/OFF		Torch 'OFF'					
Pyrometer Temp. °C			1250	1187	1126	1108	
Torch Position, in							
Torch Voltage, V/ Amps, A							
Torch Carrier Gas Pressure (PSIG)							
Center Thermocouple, °C	1158		1162	1156	1140	1116	
Furnace Pressure, in W.C.							
Notes			Note pyrometer and Tc tempera-tures are fairly close together				
Scrubber pH				1405 7.2 - 7.98 M - A			1414 7.1 - 7.85 M - A
Run time (min)	239	240					

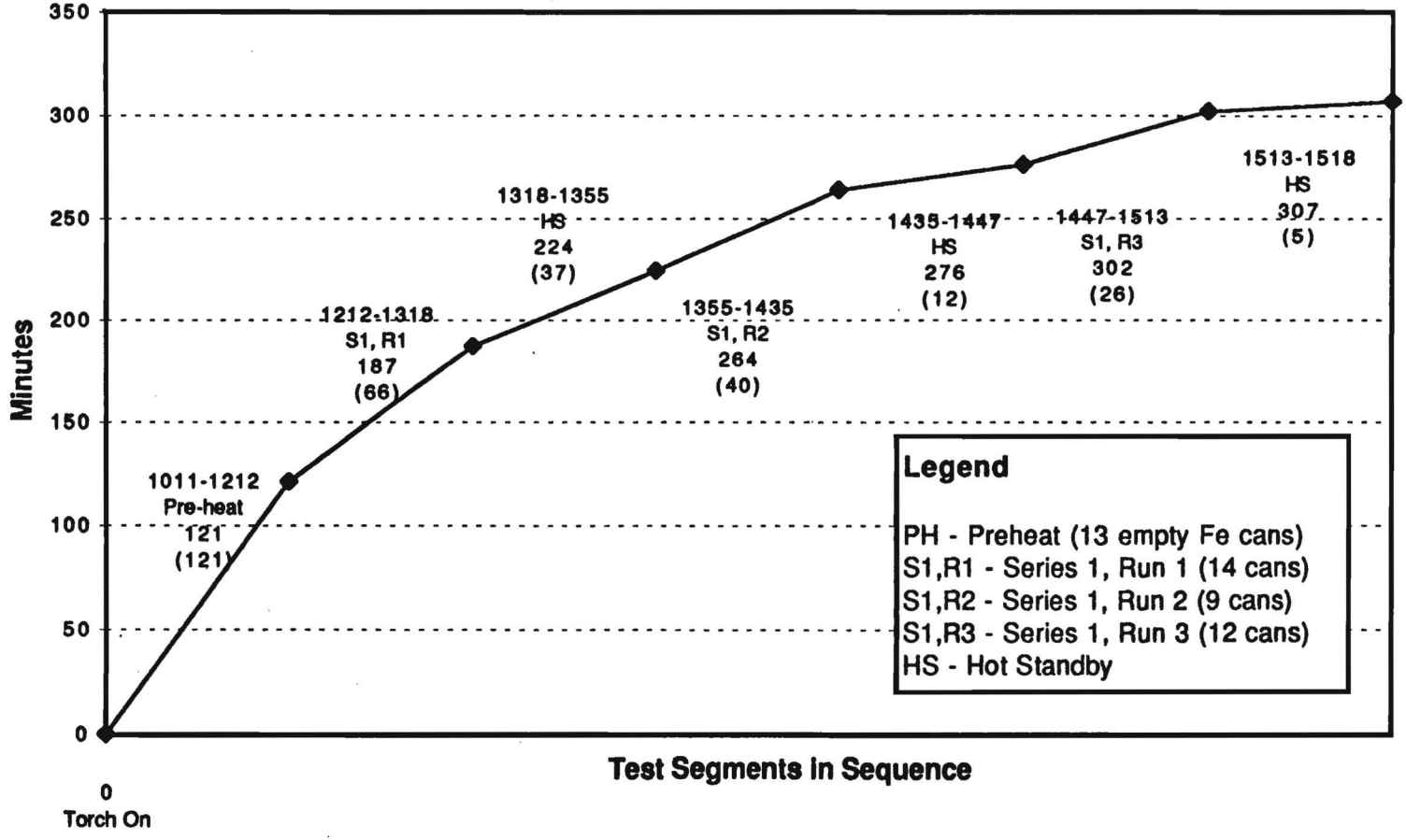
Initial: WHH
Date: 9/19/95

TEST PHASE: SERIES 2

GEORGIA TECH SURROGATE TESTING
OVERALL TEST SEGMENTS

Date	Description	Time Period	Minutes of Operation/Period
Aug. 28–Sept. 8, 1995	Cold shakedown	NA	NA
Sept. 11, 1995	Hot Shakedown	1450–1725	156
Sept. 12, 1995	Pre-heat	1011–1212	121
	Series 1, Runt 1	1212–1355	103
	Series 1, Run 2	1355–1447	52
	Series 1, Run 3	1447–1518	31
			(307 continuous)
Sept. 18 & 19 (am), 1995	Cold shakedown (w/limited torch operation outside vessel to assess air pressure)		20–30
Sept. 19, 1995	Pre-heat	1001–1156	115
	Series 2	1156–1402	126
			(241 continuous)

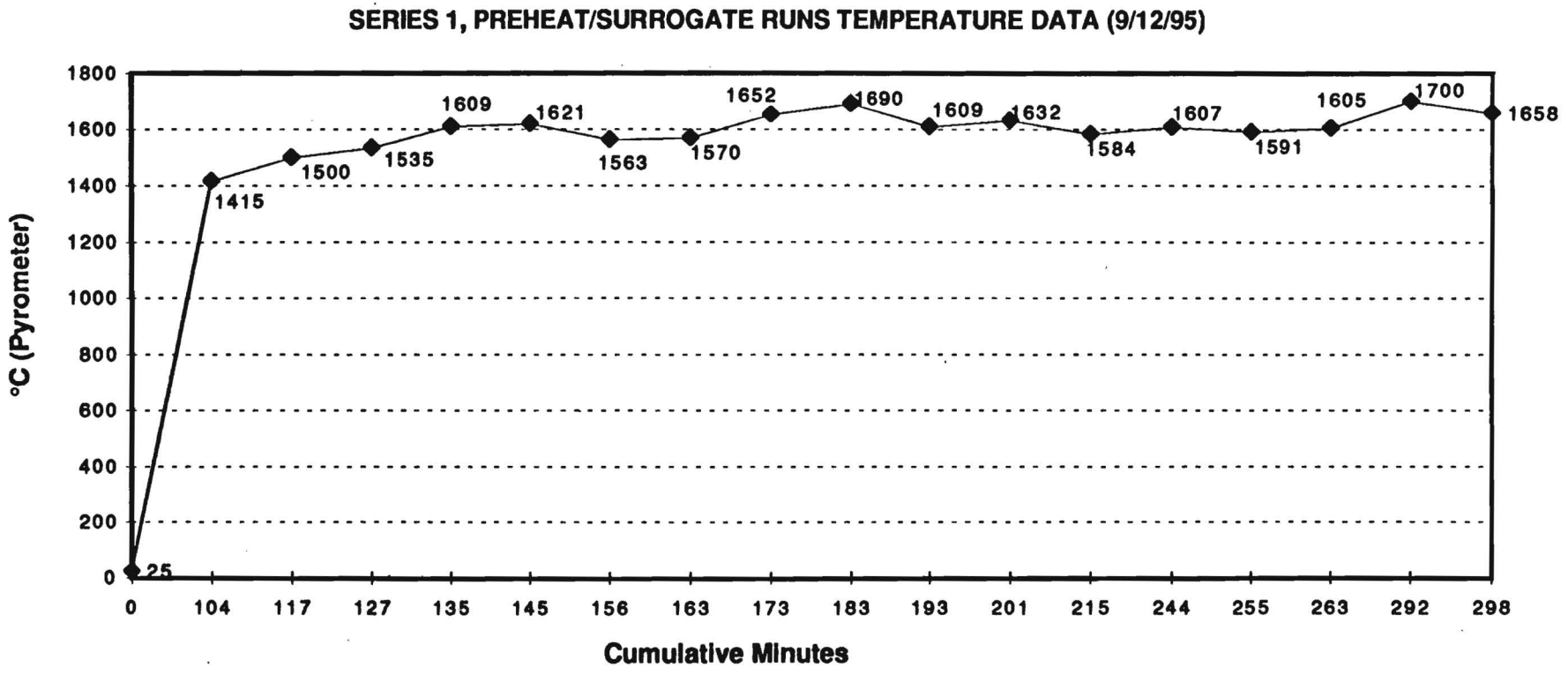
SERIES 1 TEST SEGMENTS (9/12/95)



SERIES 1, PRE-HEAT/SURROGATE RUNS

TEMPERATURE DATA (9/12/95)

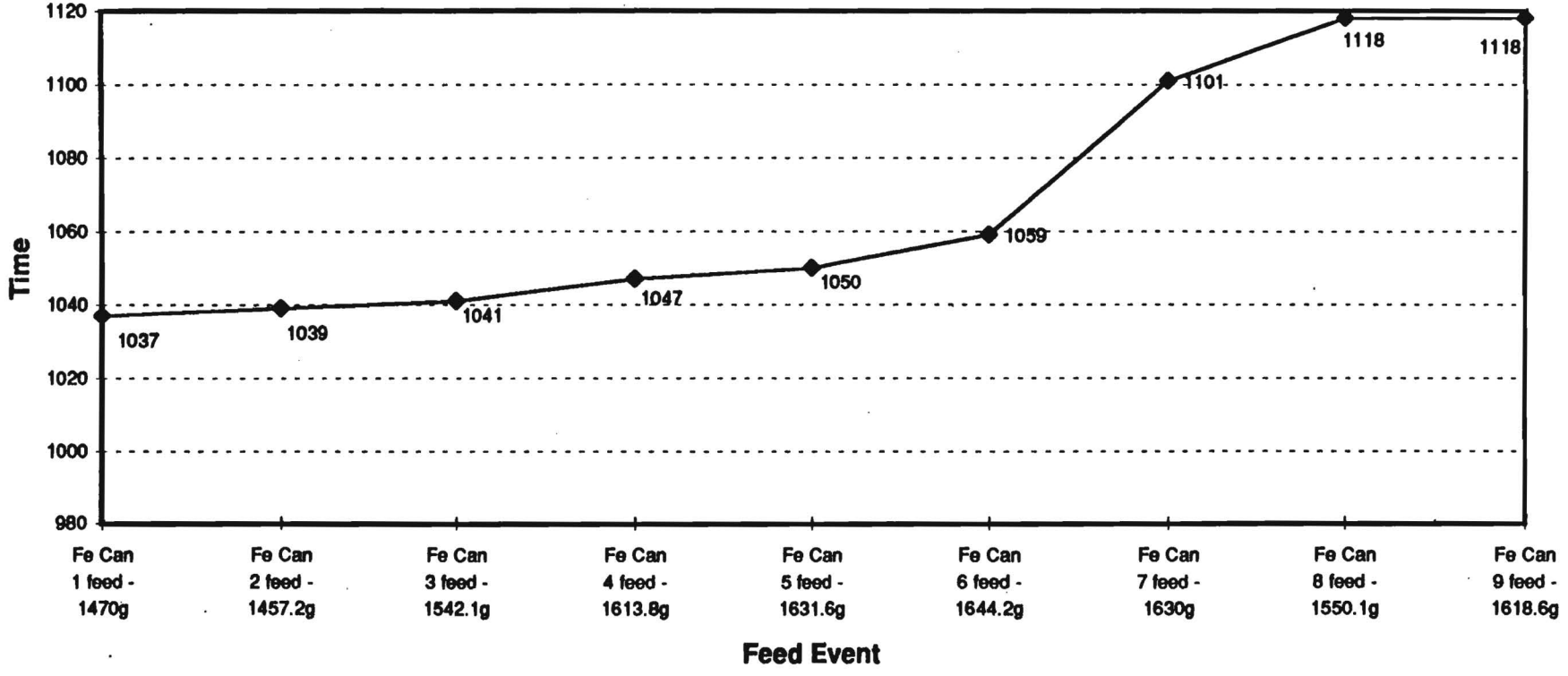
Test Segment	Time	Cum. Minutes	Infrared Pyrometer Temperature Data (°C)	Notes
Pre-Heat	1011	0	25	Assume ambient temperature at start
Pre-Heat	1155	104	1415	Consistent with hot shakedown
Pre-Heat	1208	117	1500	
Series 1, Run 1	1218	127	1535	
Series 1, Run 1	1226	135	1609	
Series 1, Run 1	1236	145	1621	
Series 1, Run 1	1247	156	1563	
Series 1, Run 1	1254	163	1570	
Series 1, Run 1	1304	173	1652	
Series 1, Run 1	1314	183	1690	
Series 1, Run 1	1324	193	1609	
Series 1, Run 1	1332	201	1632	
Series 1, Run 1	1346	215	1584	
Series 1, Run 2	1415	244	1607	
Series 1, Run 2	1426	255	1591	
Series 1, Run 2	1434	263	1605	
Series 1, Run 3	1503	292	1700	
Series 1, Run 3	1509	298	1658	

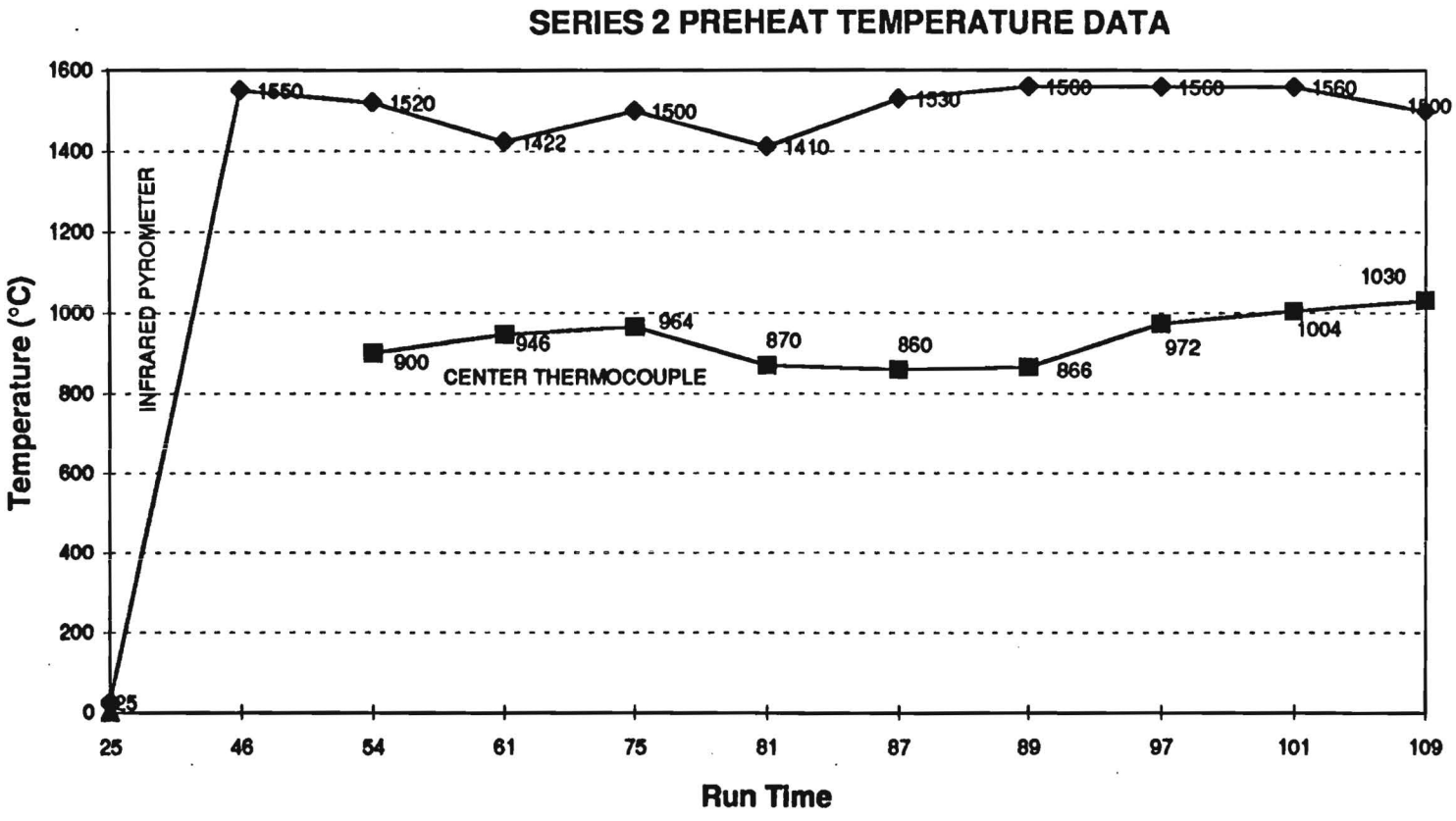

SERIES 1, PREHEAT/SURROGATE RUNS TEMPERATURE DATA (9/12/95)
°C (Pyrometer)
1800
1600
1400
1200
1000
800
600
400
200
0
25
1415
1500
1535
1609
1621
1563
1570
1652
1690
1609
1632
1584
1607
1591
1605
1700
1658
0
104
117
127
135
145
156
163
173
183
193
201
215
244
255
263
292
298
Cumulative Minutes

PRE-SERIES 2 RUN IRON ADDITION

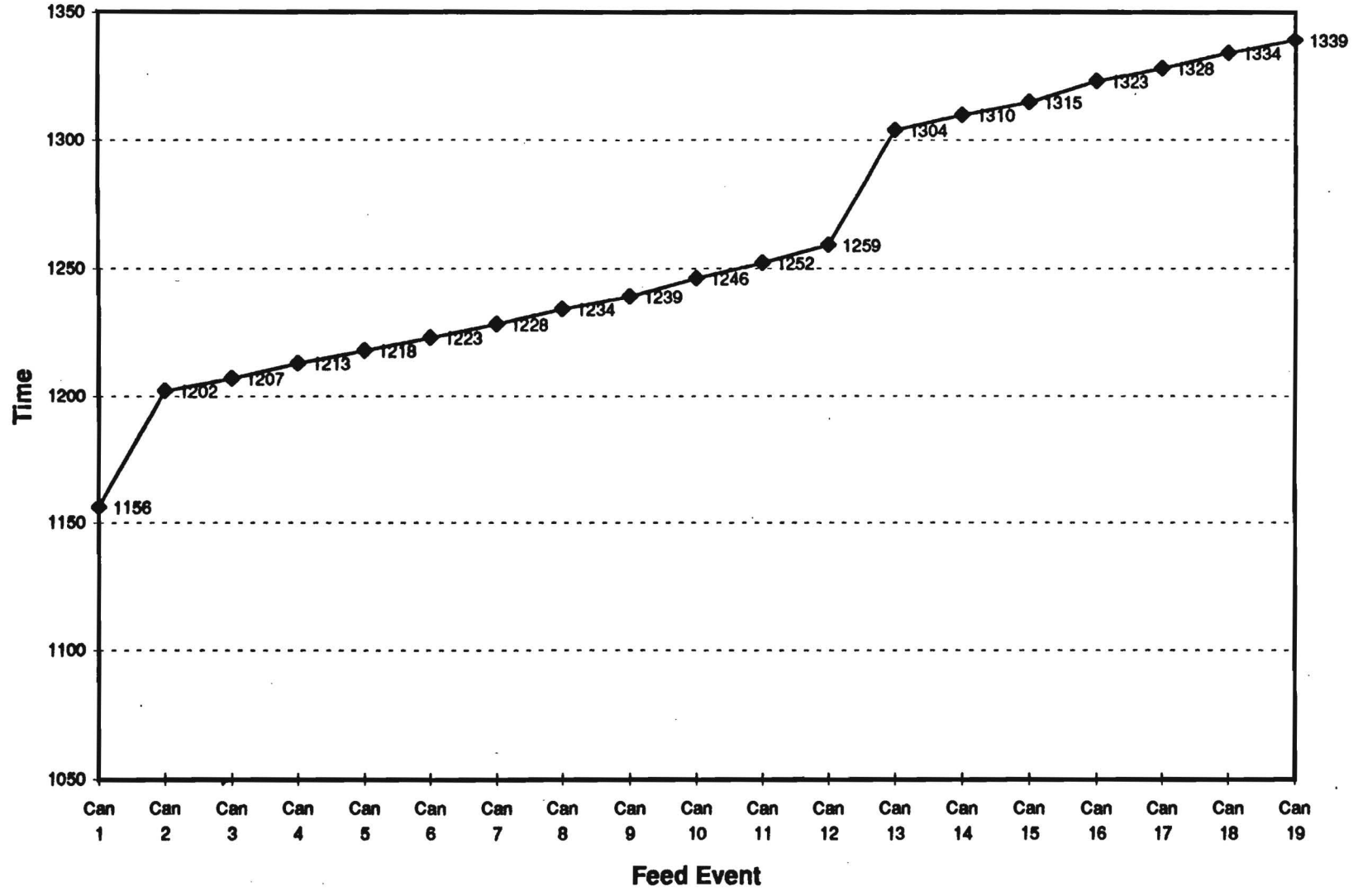
Date	Test Segment	Feed Event	Time
9/18/95	Cold Shakedown	Added 4538.4g iron to crucible (3 cans filled with washers)	
9/19/95	Pre-Heat	Iron Can 1 feed—1470 g	1037
		Iron Can 2 feed—1457.2 g	1039
		Iron Can 3 feed—1542.1 g	1041
		Iron Can 4 feed—1613.8 g	1047
		Iron Can 5 feed—1631.6 g	1050
		Iron Can 6 feed—1644.2 g	1059
		Iron Can 7 feed—1630 g	1101
		Iron Can 8 feed—1550.1 g	1118
		Iron Can 9 feed—1618.6 g	1118

PRESERIES 2 RUN IRON (Fe) ADDITION




SERIES 2 PREHEAT TEMPERATURE DATA
Temperature (°C)
Run Time
INFRARED PYROMETER
CENTER THERMOCOUPLE
1600
1400
1200
1000
800
600
400
200
0
25
46
54
61
75
81
87
89
97
101
109
1550
1520
1422
1500
1410
1530
1560
1560
1560
1500
900
946
964
870
860
866
972
1004
1030

SERIES 2 SURROGATE ADDITION (9/19/95)



TORCH POSITION ADJUSTMENT, SERIES 1

RELATIVE TO DISTANCE ABOVE CRUCIBLE INSIDE BOTTOM (9-11–12/95)

Time	Cum. Time, minutes	Torch Position, in
1450	0	12
1532	42	12
1537	47	9
1726	156	10.5
1011	0	10.5
1031	20	9
1100	49	10.5
1131	80	7
1134	83	9
1144	93	7
1147	96	9
1148	97	10.5
1325	194	9
1332	201	7
1333	202	6
1339	208	5
1343	212	4
1350	219	10.5
1447	276	11.5
1518	307	11.5

TORCH POSITION ADJUSTMENT, SERIES 2

RELATIVE TO DISTANCE ABOVE CRUICIBLE INSIDE BOTTOM, (9/19/95)

Time	Cum. Time, minutes	Torch Position, in
1001	0	9
1109	68	11
1127	86	9
1209	122	11
1409	248	11

APPENDIX E

CONTINUOUS INSTRUMENT GAS ANALYZER

Appendix E. Continuous instrument gas analyzer calibration

Gas	Parameter	Units	09/12/95	09/19/95
O_2	Upscale cal gas	vol %	10	10
	Span	vol %	25	25
	Zero gas initial response	vol %	0.1	0.13
	Deviation	% of span	0.40	0.52
	Upscale cal gas initial response	vol %	9.64	9.81
	Deviation	% of span	-1.44	-0.76
	Zero gas final response	vol %	1.65	4.71
	Deviation	% of span	6.60	18.84
O_2	Upscale cal gas final response	vol %	10.41	11.89
	Deviation	% of span	1.64	7.56
CO_2	Upscale cal gas	vol %	10	10
	Span	vol %	25	25
	Zero gas initial response	vol %	0.24	0.08
	Deviation	% of span	0.96	0.32
	Upscale cal gas initial response	vol %	10.41	9.59
	Deviation	% of span	1.64	-1.64
	Zero gas final response	vol %	1.1	0.21
	Deviation	% of span	4.40	0.84
	Upscale cal gas final response	vol %	9.68	7.24
	Deviation	% of span	-1.28	-11.04
THC	Upscale cal gas	ppmv	85.1	85.1
	Span	ppmv	100	100
	Zero gas initial response	ppmv	1.1	0.2
	Deviation	% of span	1.10	0.20
	Upscale cal gas initial response	ppmv	92.3	82.5

Appendix E. Continuous instrument gas analyzer calibration

Gas	Parameter	Units	09/12/95	09/19/95
	Deviation	% of span	7.20	-2.60
	Zero gas final response	ppmv	0.3	0.6
THC	Zero gas final response Deviation	% of span	0.30	0.60
	Upscale cal gas final response	ppmv	56.4	42.3
	Deviation	% of span	-28.70	-42.80
NO	Upscale cal gas	ppmv	8230	8230
	Span	ppmv	30000	30000
	Zero gas initial response	ppmv	-30	-30
	Deviation	% of span	-0.10	-0.10
	Upscale cal gas initial response	ppmv	8640	8490
	Deviation	% of span	1.37	0.87
	Zero gas final response	ppmv	-45	-30
	Deviation	% of span	-0.15	-0.10
	Upscale cal gas final response	ppmv	7140	4485
	Deviation	% of span	-3.63	-12.48
NO_x	Upscale cal gas	ppmv	8230	8230
	Span	ppmv	30000	30000
	Zero gas initial response	ppmv	-45	-30
	Deviation	% of span	-0.15	-0.10
	Upscale cal gas initial response	ppmv	8655	8445
	Deviation	% of span	1.42	0.72
NO_x	Zero gas final response	ppmv	-45	-30
	Deviation	% of span	-0.15	-0.10
	Upscale cal gas final response	ppmv	7185	4500
	Deviation	% of span	-3.48	-12.43

Appendix E. Continuous instrument gas analyzer calibration

Gas	Parameter	Units	09/12/95	09/19/95
CO (low)	Upscale cal gas	ppmv	450	450
	Span	ppmv	1000	1000
	Zero gas initial response	ppmv	23	7.5
	Deviation	% of span	2.30	0.75
	Upscale cal gas initial response	ppmv	482.5	456
	Deviation	% of span	3.25	0.60
	Zero gas final response	ppmv	-2.5	16.5
	Deviation	% of span	-0.25	1.65
	Upscale cal gas final response	ppmv	438.5	345.5
	Deviation	% of span	-1.15	-10.45
CO (high)	Upscale cal gas	vol %	8	8
	Span	vol %	150	150
	Zero gas initial response	vol %	0	0.325
	Deviation	% of span	0.00	0.22
	Upscale cal gas initial response	vol %	9	9
CO (high)	Upsacle cal gas initial response Deviation	% of span	0.67	0.67
	Zero gas final response	vol %	-0.275	-0.125
	Deviation	% of span	-0.18	-0.08
	Upscale cal gas final response	vol %	6.75	4.125
	Deviation	% of span	-0.83	-2.58

APPENDIX F

RESIDUALS MANAGEMENT PRODUCT MASS AND ACTIVITY BALANCE

1. BASIS MD-1 Drum

Composition (lb)		Calcines (lb)	
$TN*4H_2O$	200	ThO_2	95.65
$Ca(OH)_2$	44	CaO	33.31
PE	30		
Fe	48	Fe_2O_3	68.63
Total	322		197.59

Activity of Th-232 (nCi/g)		110
Activity of $Th\text{-}232O_2$ (nCi/g)		97
Total Activity (nCi)	4212234.7	

2. PARAMETERS

waste loading (oxide basis)	10%–100%
waste form density (g/cc)	2–4

3. PRODUCT MASS PER MD-1 DRUM

Waste Loading (%)	Additives (lb)*	Total mass (lb)	Activity (nCi/g)
10	1778.31	1975.90	4.70
20	790.36	987.95	9.39
30	461.04	658.63	14.09
40	296.39	493.98	18.78
50	197.59	395.18	23.48
60	131.73	329.32	28.17
70	84.68	282.27	32.87
80	49.40	246.99	37.56
90	21.95	219.54	42.26
100	0.00	197.59	46.96

*oxide basis

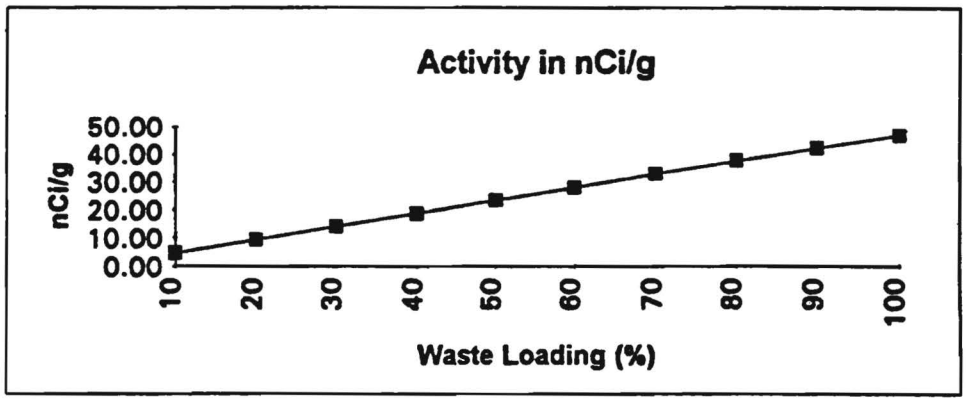
4. PRODUCT VOLUME PER MD-1 DRUM (ft^3)

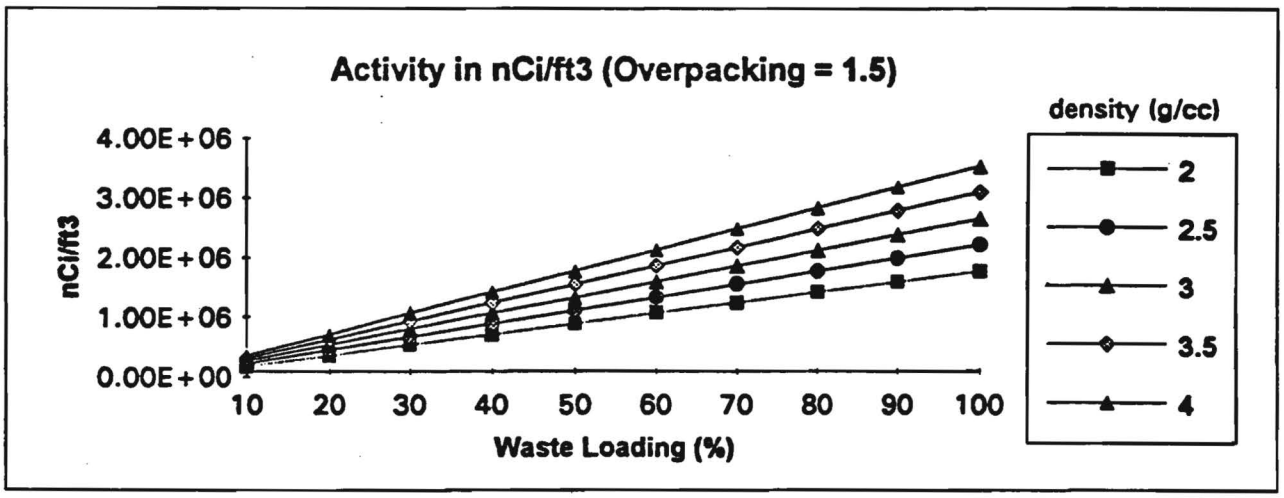
Waste Loading (%)	density (g/cc)				
	2.00	2.50	3.00	3.50	4.00
10	15.84	12.67	10.56	9.05	7.92
20	7.92	6.34	5.28	4.53	3.96
30	5.28	4.22	3.52	3.02	2.64
40	3.96	3.17	2.64	2.26	1.98
50	3.17	2.53	2.11	1.81	1.58
60	2.64	2.11	1.76	1.51	1.32
70	2.26	1.81	1.51	1.29	1.13
80	1.98	1.58	1.32	1.13	0.99
90	1.76	1.41	1.17	1.01	0.88
100	1.58	1.27	1.06	0.91	0.79

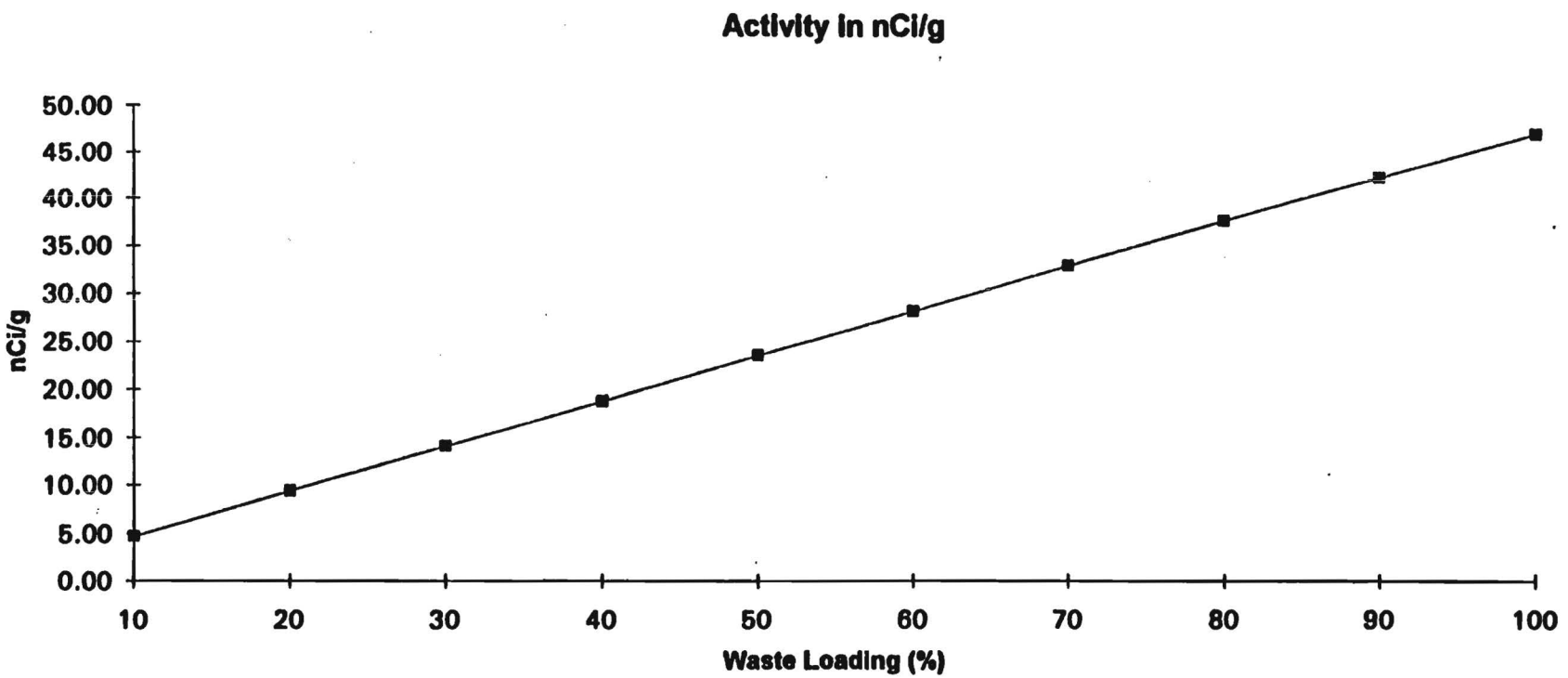
5. ACTIVITY PER UNIT VOLUME (nCi/ft³)

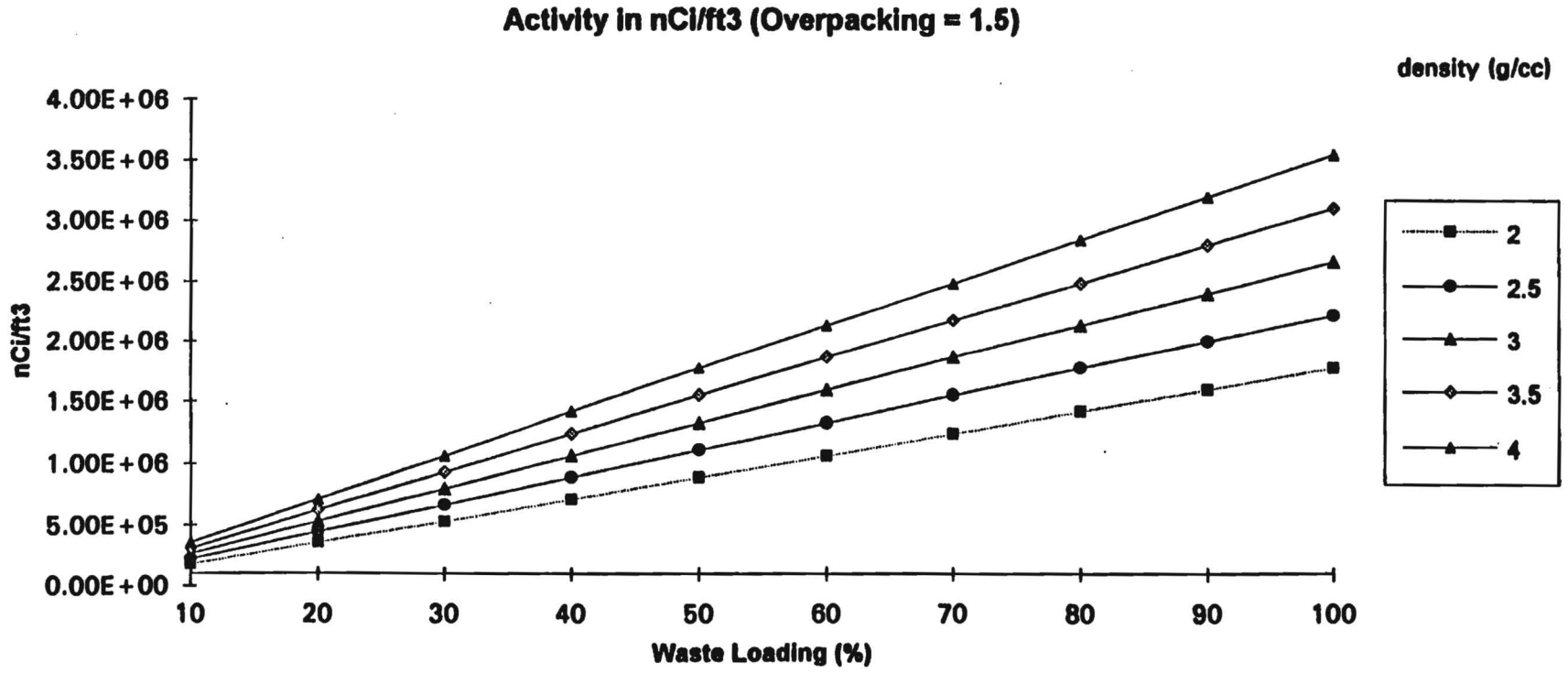
Waste Loading (%)	density (g/cc)				
	2	2.5	3	3.5	4
10	1.77E + 05	2.22E + 05	2.66E + 05	3.10E + 05	3.55E + 05
20	3.55E + 05	4.43E + 05	5.32E + 05	6.20E + 05	7.09E + 05
30	5.32E + 05	6.65E + 05	7.98E + 05	9.31E + 05	1.06E + 06
40	7.09E + 05	8.86E + 05	1.06E + 06	1.24E + 06	1.42E + 06
50	8.86E + 05	1.11E + 06	1.33E + 06	1.55E + 06	1.77E + 06
60	1.06E + 06	1.33E + 06	1.60E + 06	1.86E + 06	2.13E + 06
70	1.24E + 06	1.55E + 06	1.86E + 06	2.17E + 06	2.48E + 06
80	1.42E + 06	1.77E + 06	2.13E + 06	2.48E + 06	2.84E + 06
90	1.60E + 06	1.99E + 06	2.39E + 06	2.79E + 06	3.19E + 06
100	1.77E + 06	2.22E + 06	2.66E + 06	3.10E + 06	3.55E + 06

Waste Load	Activity (nCi/g)
10	4.70
20	9.39
30	14.09
40	18.78
50	23.48
60	28.17
70	32.87
80	37.56
90	42.26
100	46.96






Activity in nCi/g
nCi/g
50.00
45.00
40.00
35.00
30.00
25.00
20.00
15.00
10.00
5.00
0.00
10
20
30
40
50
60
70
80
90
100
Waste Loading (%)


Activity in nCi/ft3 (Overpacking = 1.5)
density (g/cc)
4.00E+06
3.50E+06
3.00E+06
2.50E+06
2.00E+06
1.50E+06
1.00E+06
5.00E+05
0.00E+00
nCi/ft3
10
20
30
40
50
60
70
80
90
100
Waste Loading (%)
2
2.5
3
3.5
4

1. BASIS MD-1 Drum (excluding Fe container)

Composition (lb)		Calcines (lb)	
$TN*4H_2O$	200	ThO_2	95.65
$Ca(OH)_2$	44	CaO	33.31
PE	30		
Fe	0	Fe_2O_3	0
Total	274		128.96

Activity of Th-232 (nCi/g)		110
Activity of Th-$232O_2$ (nCi/g)		97
Total Activity (nCi)	4212234.7	

2. PARAMETERS

waste loading (oxide basis)	10%–100%
waste form density (g/cc)	2–4

3. PRODUCT MASS PER MD-1 DRUM

Waste Loading (%)	Additives (lb)*	Total mass (lb)	Activity (nCi/g)
10	1160.64	1289.60	7.19
20	515.84	644.80	14.39
30	300.91	429.87	21.58
40	193.44	322.40	28.78
50	128.96	257.92	35.97
60	85.97	214.93	43.17
70	55.27	184.23	50.36
80	32.24	161.20	57.56
90	14.33	143.29	64.75
100	0.00	128.96	71.95

*oxide basis

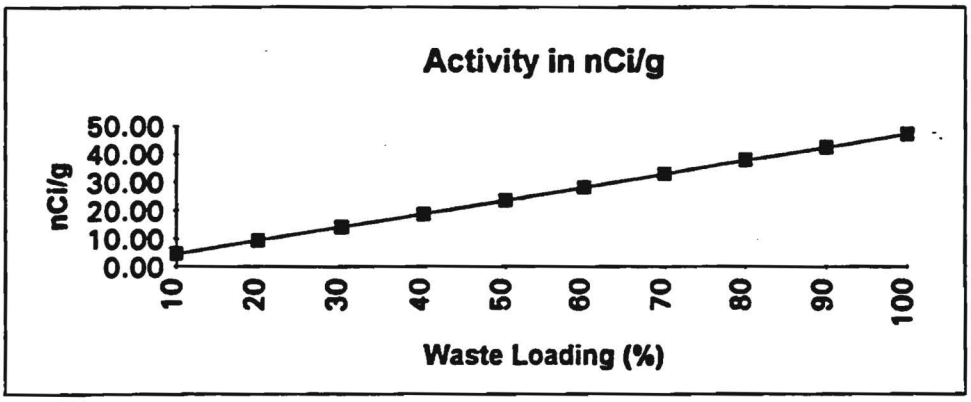
4. PRODUCT VOLUME PER MD-1 DRUM (ft^3)

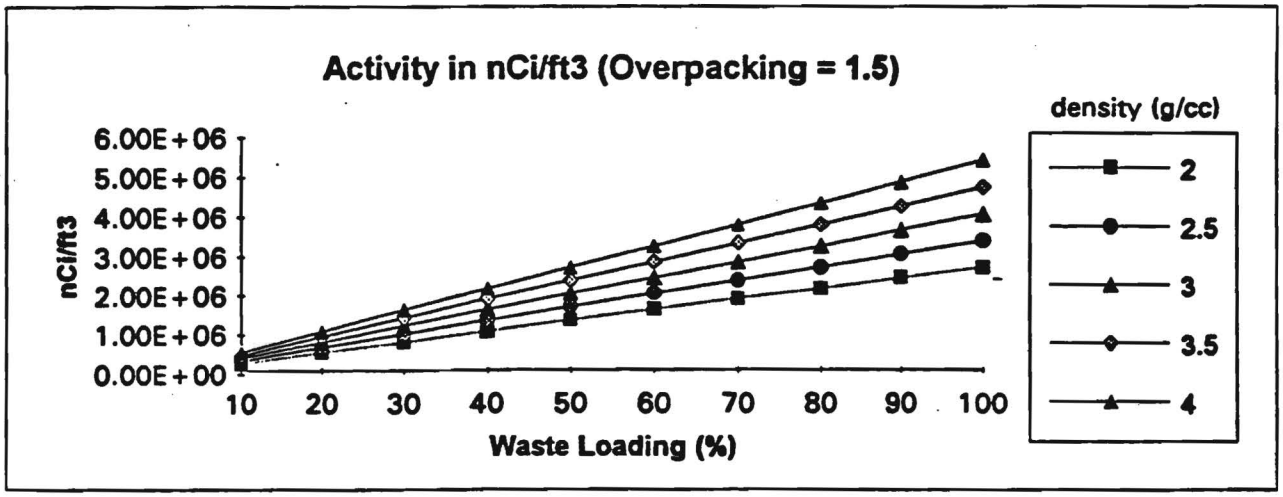
Waste Loading (%)	density (g/cc)				
	2.00	2.50	3.00	3.50	4.00
10	10.34	8.27	6.89	5.91	5.17
20	5.17	4.14	3.45	2.95	2.58
30	3.45	2.76	2.30	1.97	1.72
40	2.58	2.07	1.72	1.48	1.29
50	2.07	1.65	1.38	1.18	1.03
60	1.72	1.38	1.15	0.98	0.86
70	1.48	1.18	0.98	0.84	0.74
80	1.29	1.03	0.86	0.74	0.65
90	1.15	0.92	0.77	0.66	0.57
100	1.03	0.83	0.69	0.59	0.52

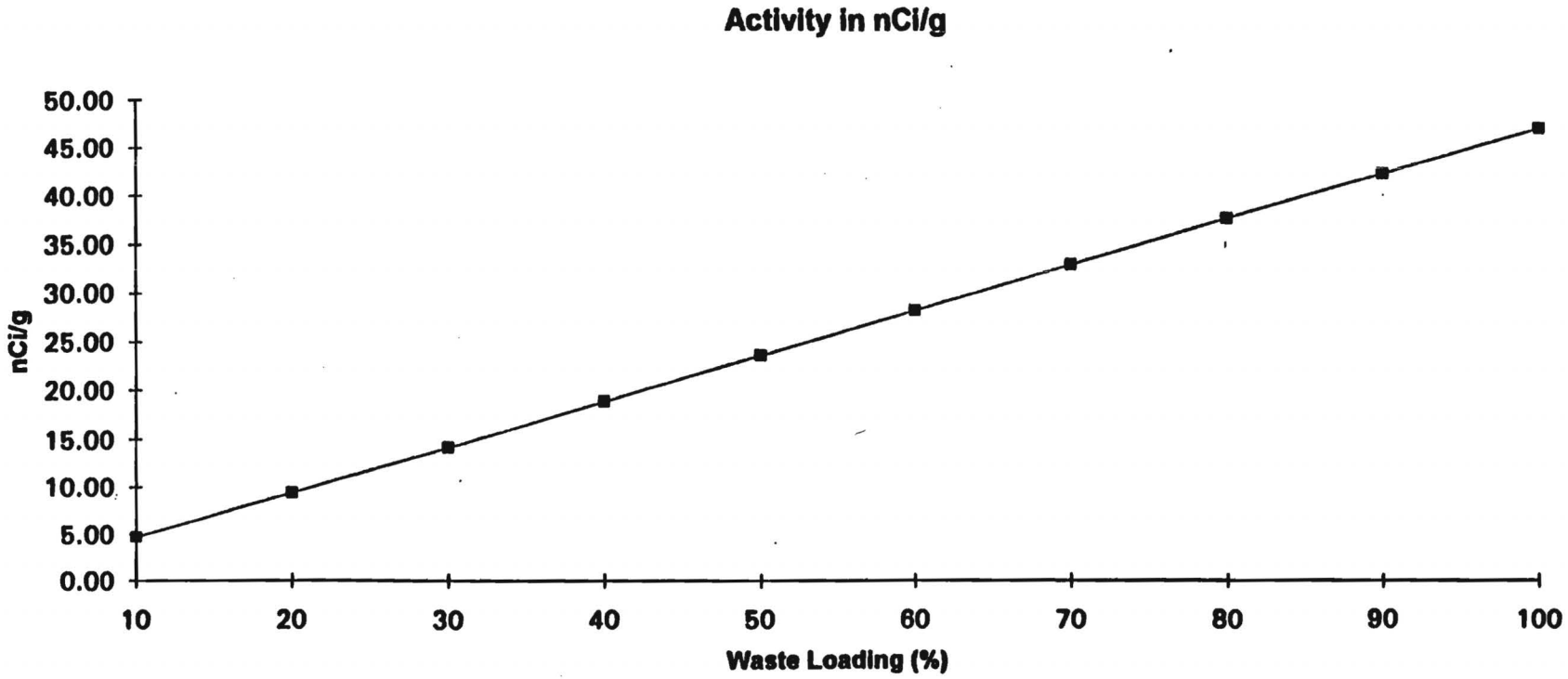
5. ACTIVITY PER UNIT VOLUME (nCi/ft³)

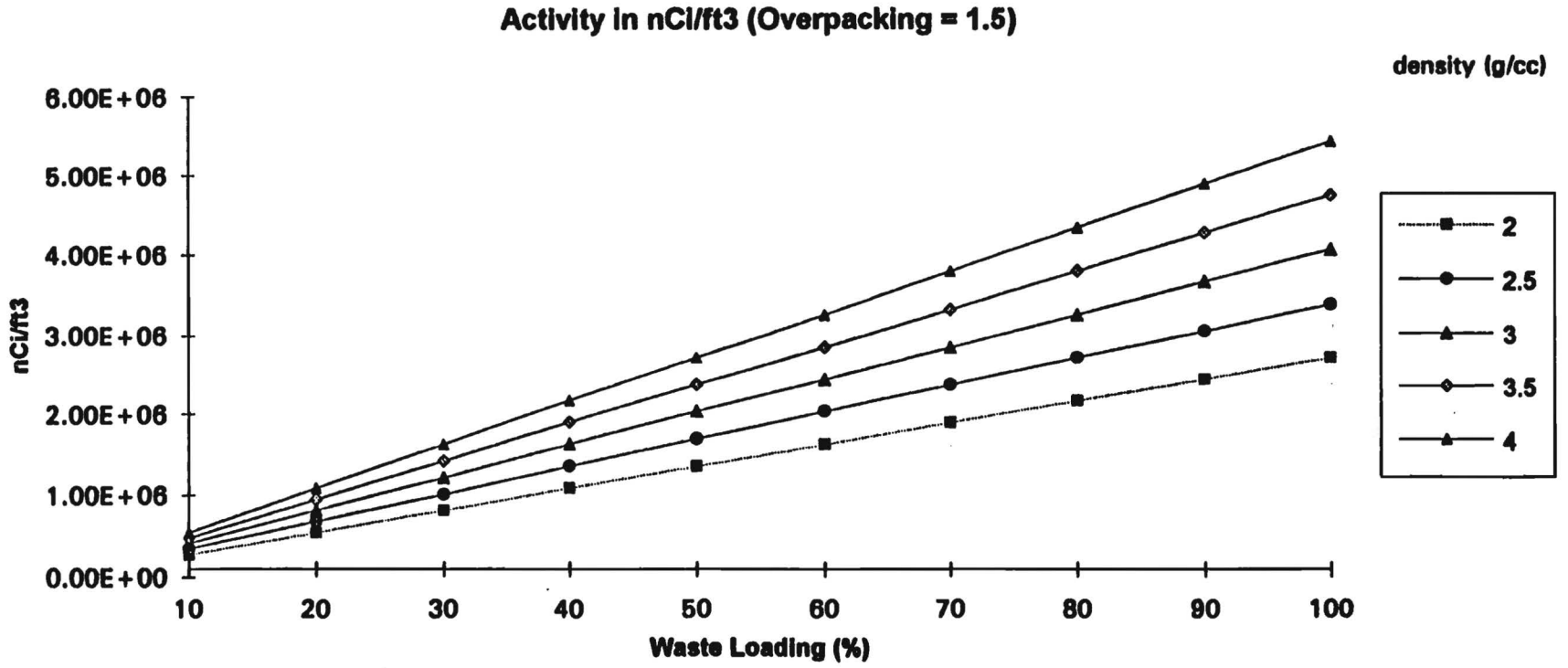
Waste Loading (%)	density (g/cc)				
	2	2.5	3	3.5	4
10	2.72E + 05	3.40E + 05	4.07E + 05	4.75E + 05	5.43E + 05
20	5.43E + 05	6.79E + 05	8.15E + 05	9.51E + 05	1.09E + 06
30	8.15E + 05	1.02E + 06	1.22E + 06	1.43E + 06	1.63E + 06
40	1.09E + 06	1.36E + 06	1.63E + 06	1.90E + 06	2.17E + 06
50	1.36E + 06	1.70E + 06	2.04E + 06	2.38E + 06	2.72E + 06
60	1.63E + 06	2.04E + 06	2.44E + 06	2.85E + 06	3.26E + 06
70	1.90E + 06	2.38E + 06	2.85E + 06	3.33E + 06	3.80E + 06
80	2.17E + 06	2.72E + 06	3.26E + 06	3.80E + 06	4.35E + 06
90	2.44E + 06	3.06E + 06	3.67E + 06	4.28E + 06	4.89E + 06
100	2.72E + 06	3.40E + 06	4.07E + 06	4.75E + 06	5.43E + 06

Waste Load	Activity (nCi/g)
10	4.70
20	9.39
30	14.09
40	18.78
50	23.48
60	28.17
70	32.87
80	37.56
90	42.26
100	46.96






Activity in nCi/g
50.00
45.00
40.00
35.00
30.00
25.00
20.00
15.00
10.00
5.00
0.00
nCi/g
10
20
30
40
50
60
70
80
90
100
Waste Loading (%)


Activity in nCi/ft3 (Overpacking = 1.5)
density (g/cc)
6.00E+06
5.00E+06
4.00E+06
3.00E+06
2.00E+06
1.00E+06
0.00E+00
nCi/ft3
10
20
30
40
50
60
70
80
90
100
Waste Loading (%)
2
2.5
3
3.5
4